FACTORS INFLUENCING THE STRENGTH DEVELOPMENT IN PFA-LIME SYSTEMS

by

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VOLUME II

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CHAPTER ONE

TABLES, FIGURES AND PLATES

Table 1.1

Production, sales and disposal figures of the pfa produced in U.K. between 1976/7 to 1986/7. [10]

(Mte)	1976/7	1977/8	1978/9	1979/80	1980/1	1981/2	1982/3	1983/4	1984/5	1985/6	1986/7
Production (Dry)	12.40	12.34	13.51	14.80	14.07	13.16	12.87	13.04	6.68	12.64	12.55
ASH USE (Dry and Damp)											
(a) Sales	3.91	3.96	4.80	4.52	3.76	4.70	4.38	4.70	3.55	4.68	5.24
(b) CEGB Works	1.10	1.12	1.37	1.61	1.59	1.52	1.49	1.33	0.29	1.05	1.03
ASH DISPOSAL	7.39	7.26	7.34	8.67	8.72	6.93	7.01	7.01	3.69	6.91	6.28

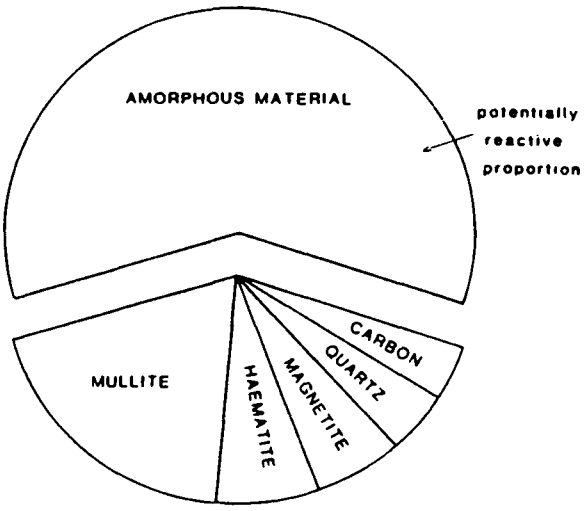
Table 1.2

Selected national standard specifications for pfa for use in concrete (after Dhir [4]).

Requirement	Australia	Austria	Canada Class F	Canada Class C	India	Japan	Korea	Turkey	UK	USA Class F	USA Class C	USSR
Loss-on-ignition, max %	8.0	5.0	12.0	6.0	12.0	5.0	12.0	10.0	7.0	12.0	6.0	10.0
SO_3, max %	2.5	—	5.0	5.0	3.0	—	5.0	5.0	2.5	5.0	5.0	3.0
MgO, max %	—	5.0	—	—	5.0	5.0	5.0	5.0	4.0	5.0	5.0	—
Available alkali metal as Na_2O, max %	—	—	—	—	1.5	—	—	—	—	1.5	1.5	—
SiO_2, min %	—	42/60	—	—	35	45	—	—	—	—	40	—
$SiO_2 + Al_2O_3 + Fe_2O_3$, min %	—	—	—	—	70	70	70	70	—	70	50	—
Moisture, max %	1.5	1.0	3.0	3.0	—	1.0	3.0	3.0	0.5	3.0	3.0	—
Water requirement, max %	—	—	—	—	—	100	102	105	95	105	105	—
Pozzolanic activity index:												
with cement, min %	—	80	75	75	—	60/70	85	70	85	75	75	85
with lime, min N mm^{-2}	-	—	—	—	3.9	-	5.5	-	—	5.5	5.5	5.5
Fineness												
Ret. on 45 μm sieve, max %	50	—	34	34	—	—	—	—	12.5	34	34	—
Specific surface m^2 kg min			—	—	320	270	—		-			-

Table 1.3
Typical chemical compositions of the pfa produced in U.K. [11].

	Per cent		
	Max.	*Min.*	*Typical*
Silicon (as SiO_2)	51	45	48
Aluminium (as Al_2O_3)	32	24	27
Iron (as Fe_2O_3)	11	7	9
Calcium (as CaO)	5.4	1.1	3.3
Magnesium (as MgO)	4.4	1.5	2.0
Potassium (as K_2O)	4.5	2.8	3.8
Sodium (as Na_2O)	1.7	0.9	1.2
Titanium (as TiO_2)	1.1	0.8	0.9
Sulphur (as SO_3, soluble)	1.3	0.3	0.6
Chlorine (as Cl)	0.15	0.05	0.08



Phase	Mean (%)	Min. (%)	Max. (%)
Amorphous	59	30	78
Mullite	19	7	46
Haematite	7	2	15
Magnetite	6	2	10
Quartz	5	1	12
Carbon	4	1	13

Figure 1.1 Typical phase composition of the pfa produced in U.K. (after Dhir [4]).

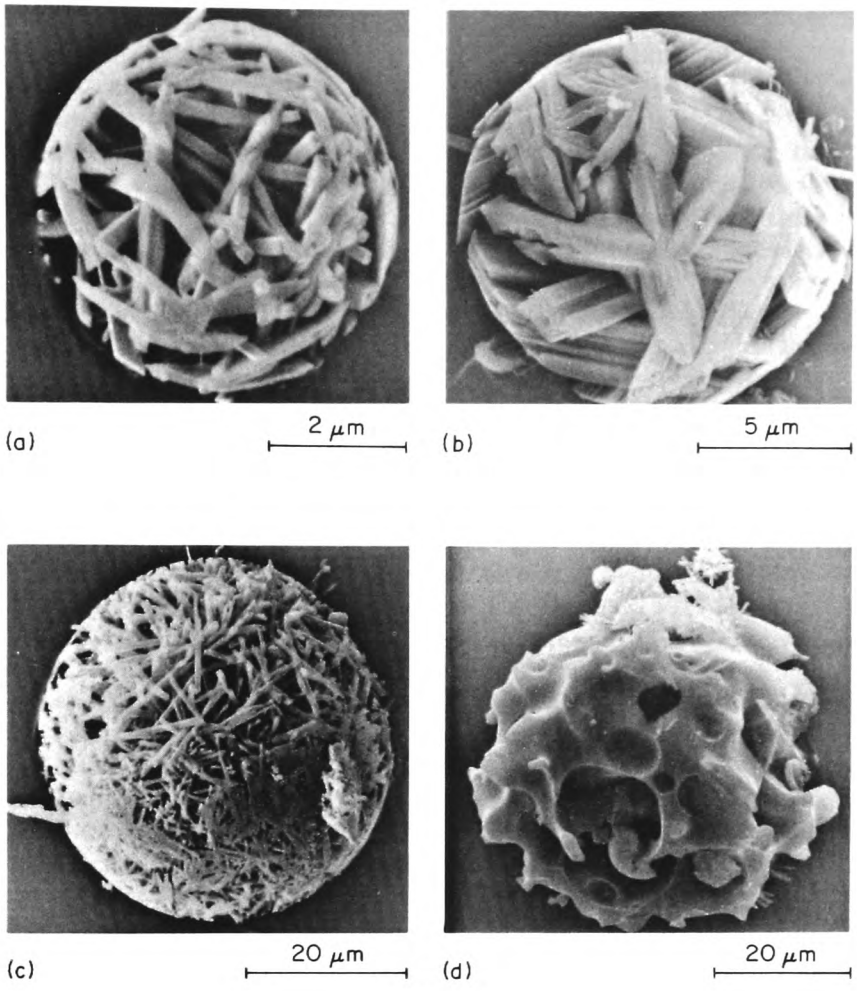
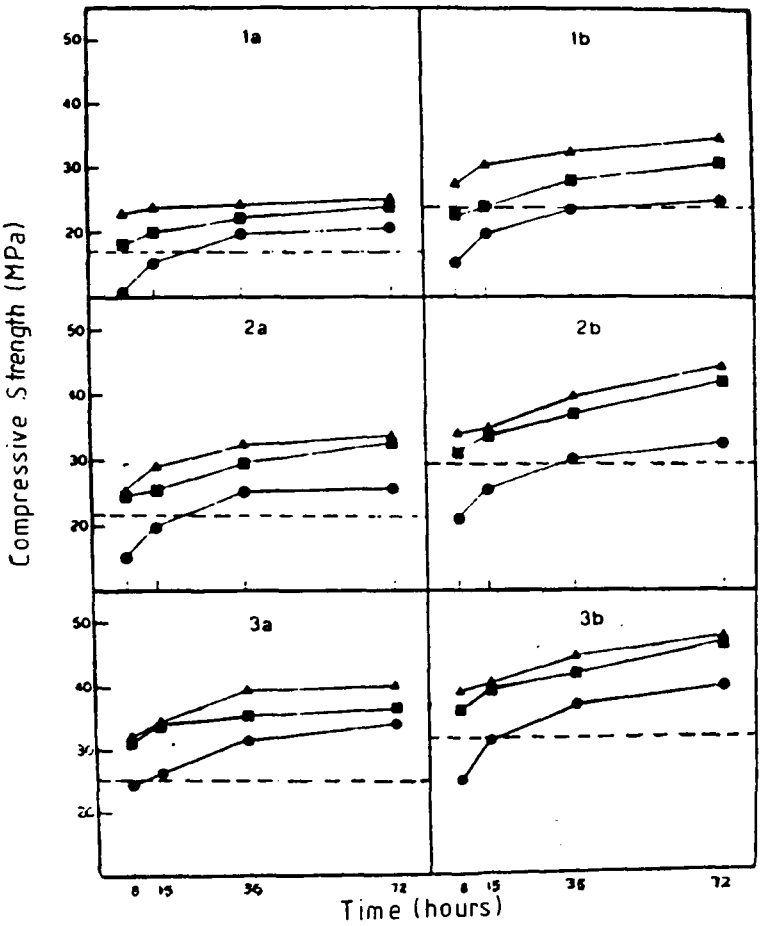


Figure 1.2 Mullite and quartz skeletons of pfa particles after etching with 1% HF solutions (after Hullett and Weinberger [21]).



Compressive strength as a function of curing time. a. compaction pressure = 50 MPa, b. compaction pressure = 150 MPa. 1. $Ca(OH)_2$ 20%, fly ash 80%; 2. $Ca(OH)_2$ 30%, fly ash 70%; 3.$Ca(OH)_2$ 40%, fly ash 60%. ● : 60°C; ■ : 75°C; ▲ :90°C. ------- Specimen treated for 28 days at 25°C.

Figure 1.3 Compressive strength results of pfa-lime compacts reported by Marcialis et.al.[25].

CHAPTER TWO

TABLES, FIGURES AND PLATES

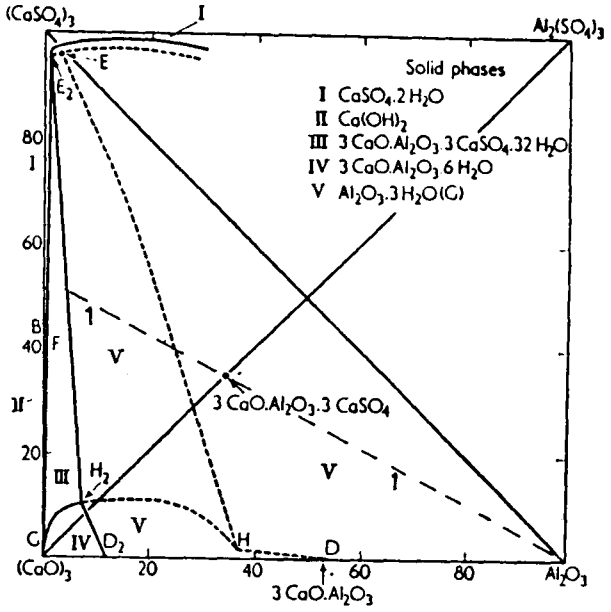


Figure 2.1 System $CaO-Al_2O_3-CaSO_4-H_2O$ at 25°C (after Turriziani [29] and Jones [31]).

CHAPTER THREE

TABLES, FIGURES AND PLATES

Table 3.1

List of the experimental techniques used and the nature of the results obtained.

Technique	Nature of results obtained
X-ray powder diffraction (XRD)	Identification of the crystalline reaction products present and semi-quantitative assessment of the relative proportions in which they were present.
Differential thermogravimetry (DTG)	Quantitative determination of the lime consumption during reaction, characterisation of both crystalline and non-crystalline reaction products with some semi-quantitative assessment of the amounts formed
Scanning electron microscopy (SEM)	Characterisation and identification of both amorphous and crystalline reaction products, including morphological development and semi-quantitative analysis.
Transmission electron microscopy (TEM)	Characterisation of reaction products (particularly amorphous products) and detailed composition analysis of these products.
Mercury intrusion porosimetry (MIP)	Quantitative measurements of the changes in porosity and pore size distribution of the cured samples.
Permeability test	Quantitative measurements of the permeabilities of the cured samples.

Table 3.2

Summary of the thermal analysis data observed by other workers for compounds relevant to the current work.

Phase	Technique	Dehydration Temperature at Peak °C	Number of Peaks	Ref.
1. Gel phases				
CSH gel	DTA	140	1	27
CSH gel	TG	120-140	1	77
CSSH gel	DTA	180	1	78
CASH gel	DTG	120-140 & 350	2	79
AH_3 gel	DTG	121	1	80
AH_3 gel	DTA	100-108	1-2	81
2. Calcium aluminate hydrate phases				
C_4AH_{13}	DTA	220 & 290	2	82
C_2AH_8	DTA	164-192	1	81
C_2AH_8	DTA	194	1	80
C_4AH_{13} & C_2AH_8	DTG	120, 200 & 280	3	40
C_4AH_{13} & C_2AH_8	DTA	120, 210 & 290	3	83
C_3AH_6	DTA	315-330	1	82
C_3AH_6	DTG	320	1	40
C_3AH_6	DTA	330	1	83
C_3AH_6	DSC	300	1	58
C_3AH_6	DTA	304-333	1	81
C_3AH_6	DTA	300-324	1	80

Table 3.2 continued...

3. Calcium sulphoaluminate phases				
$C_3A.3C\underline{S}.H_{32}$	DTA	160 & 270-300	2	82
$C_3A.3C\underline{S}.H_{32}$	DTA	160	1	27
$C_3A.3C\underline{S}.H_{32}$	DTG	120	1	40
$C_3A.3C\underline{S}.H_{32}$	DTA	140	1	83
$C_3A.3C\underline{S}.H_{32}$	DTA	140	1	84
$C_3A.3C\underline{S}.H_{32}$	DSC	80-125	1	58
$C_3A.3C\underline{S}.H_{32}$	DSC	90-110	1	22
$C_3A.3C\underline{S}.H_{32}$	DTA	78-135	1	85
$C_3A.3C\underline{S}.H_{32}$	TG	70-110	1	46
$C_3A.3C\underline{S}.H_{32}$	DTG	92, 280 & 370	3	*
$C_3A.C\underline{S}.H_{12}$	DTA	220	1	27
$C_3A.C\underline{S}.H_{12}$	DTG	220	1	40
$C_3A.C\underline{S}.H_{12}$	DSC	190	1	58

* - Determined in this work (see Section 4.4 in Chapter 4).

4. Gypsum				
$C\underline{S}H_2$	DTG	150 & 160	2	40
$C\underline{S}H_2$	DTA	160	1	83
$C\underline{S}H_2$	DTA	160	1	84
$C\underline{S}H_2$	DSC	135-160	sometimes a doublet	58
$C\underline{S}H_2$	DTG	182	1	**

** - Determined in this work (see Section 4.2 in Chapter 4).

Table 3.2 continued...

5. Aluminium hydroxide				
γAH_3	DTA	272-295	1	81
γAH_3	DTA	296	1	80

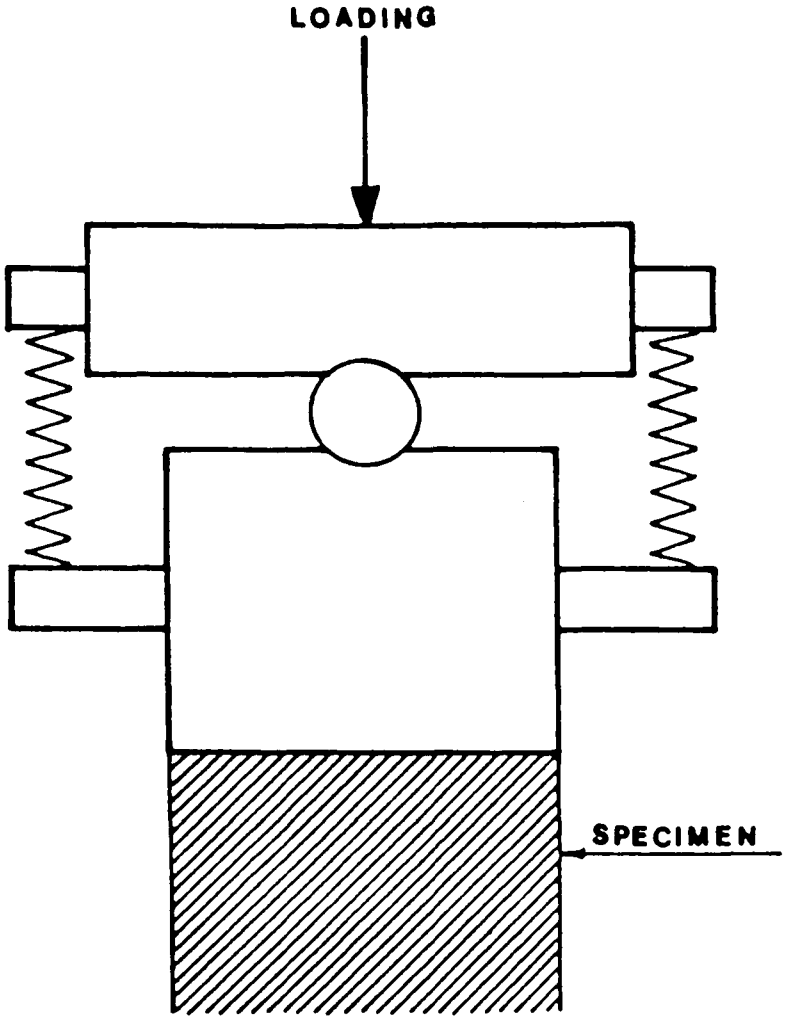


Figure 3.1 Schematic representation of the self-levelling cap used for determination of the unconfined compressive strength. (after Arabi [72]).

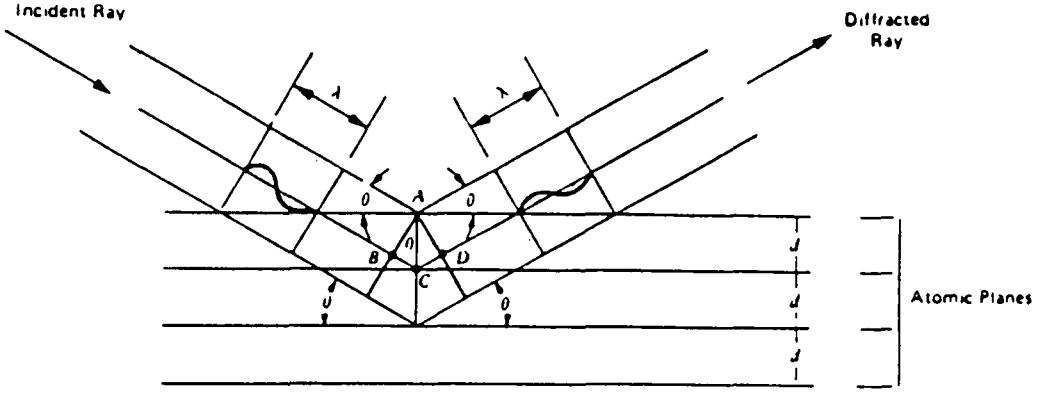
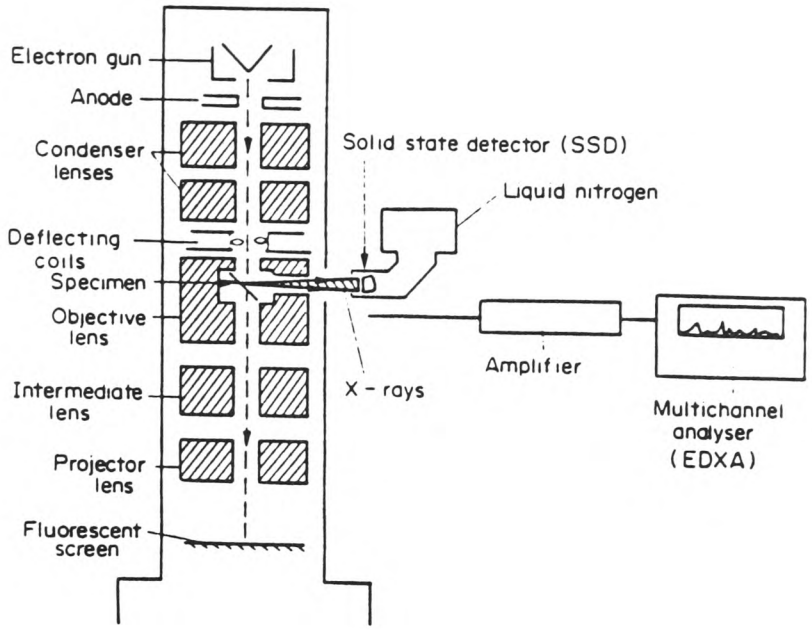
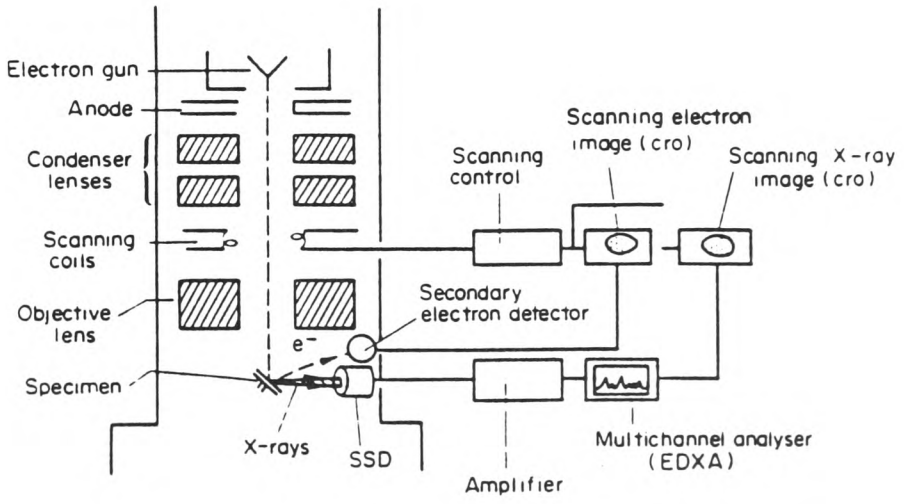


Figure 3.2 Geometrical conditions for X-ray diffraction according to Bragg's law. (after Mitchell [73]).

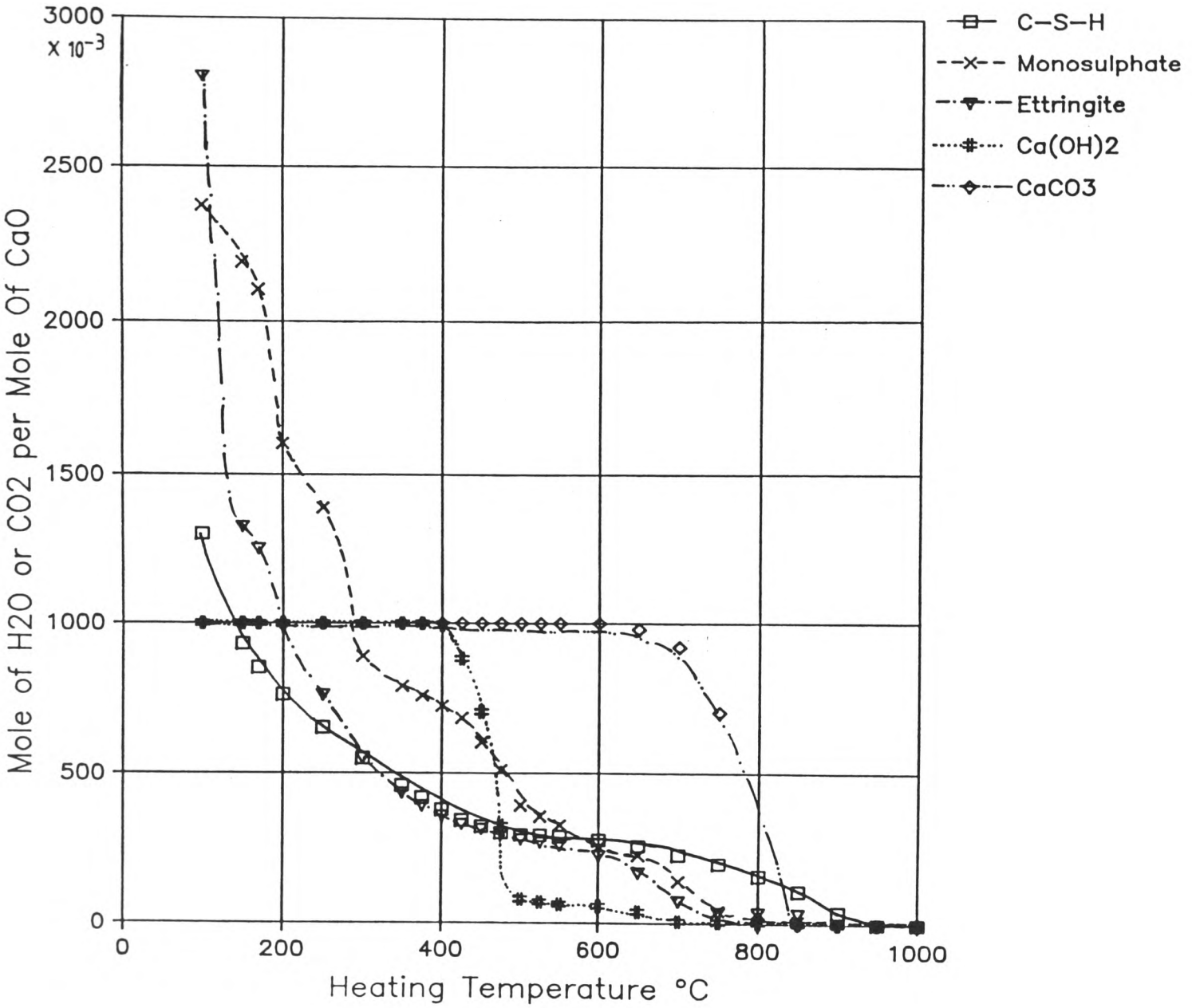


(a)



(b)

Figure 3.3 Schematic representation of the systems employed for a) Transmission Electron Microscopy (TEM) and b) Scanning Electron Microscopy (SEM). (after Sarkar [74]).


3000
X 10⁻³
2500
2000
1500
1000
500
0
Mole of H2O or CO2 per Mole Of CaO
0
200
400
600
800
1000
Heating Temperature °C
C–S–H
Monosulphate
Ettringite
Ca(OH)2
CaCO3

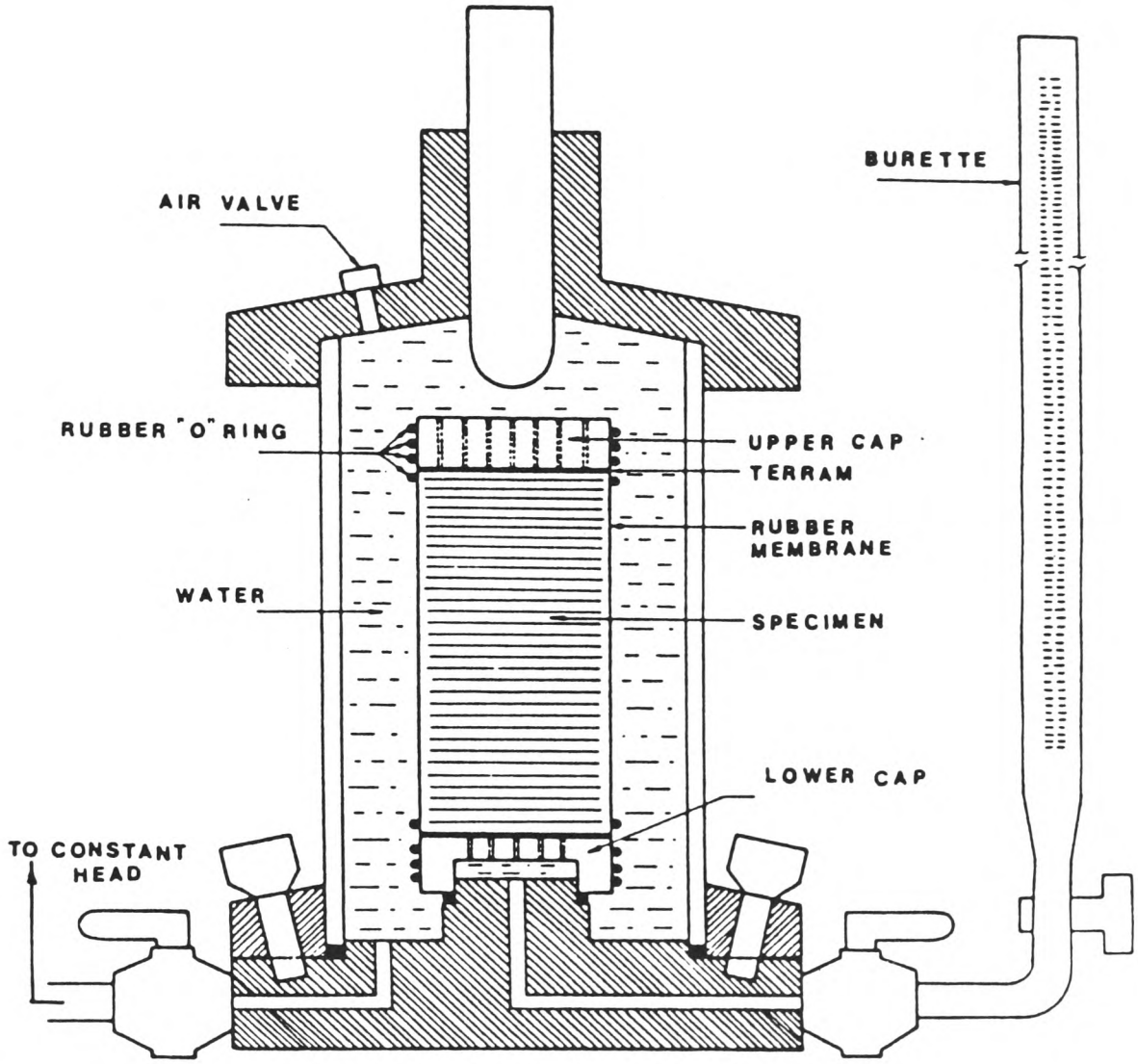


Figure 3.5 Schematic representation of the apparatus used for measurement of the permeability of the cured pfa-lime specimens. (After Arabi [72]).

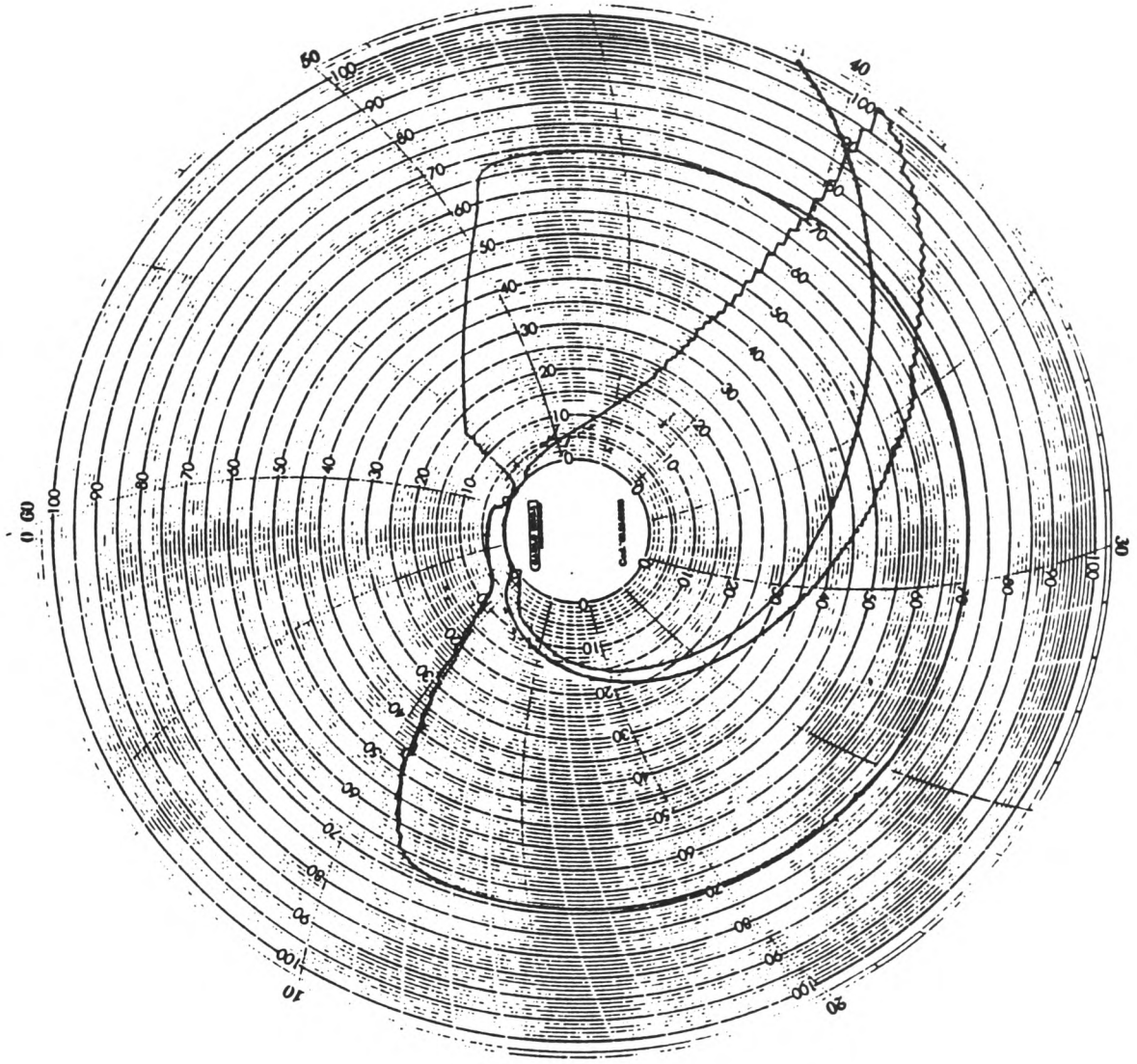


Figure 3.6 Typical output from the Carlo Erba porosimeter, the sample was pfa + 20wt.% lime cured for 14 days at 95°C and 100% r.h.

Plate 3.1 Photograph showing the equipment used in sample preparation.

CHAPTER FOUR

TABLES, FIGURES AND PLATES

Table 4.1

Chemical and grading analyses of the lime used [94].

Typical chemical analysis

			%
Calcium hydroxide	$Ca(OH)_2$		96.79
Calcium carbonate	$CaCO_3$		1.36
Calcium sulphate	$CaSO_4$		0.06
Magnesium oxide	MgO		0.83
Ferric oxide	Fe_2O_3		0.06
Aluminium oxide	Al_2O_3		0.10
Silica	SiO_2		0.46
Excess moisture	H_2O		0.34
Neutralising value	($\equiv$ CaO)	Minimum	74

Typical grading analysis

	% Passing
Nominal aperture sizes (microns)	
500	100
355	99.99
250	99.98
180	99.93
125	99.74
90	99.59
63	94.71
Specific surface area m^2/kg	1300–1500

Table 4.2

Chemical composition of the pfa used. (provided by the supplier Pozzolanic Lytag Ltd.)

ELEMENT	% COMPOSITION
Silica as SiO_2	47.7
Alumina as Al_2O_3	31.6
Iron as Fe_2O_3	10.0
Calcium as CaO	2.2
Magnesium as MgO	1.3
Sodium as Na_2O	1.1
Potassium as K_2O	4.2
Titanium as TiO_2	1.0
Sulphate as SO_3	0.88

Table 4.3

The concentrations of Na, K and Ca in solution in pfa-distilled water suspensions after boiling, determined using flame photometry technique.

Element	Solvent*	Concentration (ppm)	Concentration percent as Oxide**
Na	water	14.4	0.00776
K	water	9.2	0.00443
Ca	water	210	0.118

* - solution from 600 g of pfa + 2400 ml of boiled distilled water.
** - expressed as percentage by weight of pfa.

Table 4.4

XRD results for the pfa, (a) in its as supplied "dry" state, and (b) after being moistened with 11wt.% of distilled water.

(a) d	(a) I	(a) C	(b) d	(b) I	(b) C
			0.960	vw	E
0.760	w	G	0.756	m	G
0.539	w	M	0.539	w	M
0.426	w	Q+G	0.426	w	Q+G
			0.378	m	G
			0.374	m	G
0.343	w	M	0.341	w	M
0.339	m	M	0.338	w	M
0.335	s	Q	0.334	m	Q
			0.305	w	G
0.295	vw	Mg			
0.288	vw	M	0.287	vw	M
0.269	w	M+Ha	0.268	w	M+Ha
0.254	w	M+Mg	0.254	w	M+Mg

Keys : d - d spacings (nm), I - peak intensity, C - compound, E - ettringite, G - gypsum, M - mullite, Q - quartz, Mg - magnitite, Ha - haematite.

Table 4.5

The concentrations of Na, K, and Ca in solution in acidified pfa-distilled water suspension after boiling determined using flame photometry technique.

Element	Solvent*	Concentration (ppm)	Concentration percent as Oxide**
Na	dilute HCl	23	0.018
K	dilute HCl	23	0.011
Ca	dilute HCl	588	0.334

* - solution from 600 g of pfa + 2400 ml of boiled distilled water + 35 ml of HCl.
** - expressed as percentage by weight of pfa.

Table 4.6

Experimental and theoretical values of percentage weight loss of natural ettringite on ignition (number of water molecules represented by the weight losses are given in brackets).

	experimental	theoretical
% weight loss at 92°C	26.6 (19)	37.3 (26)
% weight loss at 280°C	12.6 (9)	8.6 (6)
% weight loss at 355°C	4.02 (3)	-
% total weight loss	43.4 (31)	45.9 (32)

Table 4.7

XRD results for mixtures of pfa + 20wt.% added lime when a) in the as supplied "dry" state, and b) after being moistened with 30.2wt.% (of the weight of the pfa) of distilled water.

(a)			(b)		
d	I	C	d	I	C
			0.556	w	E
0.538	w	M	0.535	mw	M
0.488	vs	L	0.488	s	L
			0.469	w	E
0.425	mw	Q	0.424	mw	Q
			0.389	mw	E
0.341	m	M	0.341	m	M
0.338	m	M	0.339	m	M
0.335	ms	Q	0.334	ms	Q
0.309	m	L	0.309	ms	L
0.302	w	Ca	0.302	m	Ca
0.295	w	Mg			
0.287	w	M	0.287	mw	M
0.269	mw	M+Ha	0.268	m	M+Ha
0.261	vvs	L	0.262	vs	L
0.253	w	M+Mg	0.254	mw	M+Mg
0.251	mw	Ha+Ca	0.251	mw	Ha+Ca
0.229	vw	Ca	0.228	w	Ca
0.220	w	M+Ha	0.220	mw	M+Ha
0.192	m	L	0.192	m	L

Keys : d - d spacings (nm), I - peak intensity, C - compound, L - lime, Ca - calcite, E - ettringite, M - mullite, Q - quartz, Mg - magnetite, Ha - haematite.

Table 4.8

Comparison of the observed weight losses at 73°C and 350 °C with the calculated weight loss for ettringite dehydration, assuming that the 1.34% gypsum present in the pfa forms ettringite.

Theoretical ettringite content (if all the gypsum present forms crystalline ettringite)		3.26%
Theoretical weight loss on dehydration	(26 H_2O)	1.22%
	(6 H_2O)	0.28%
	total	1.50%
Observed weight loss (determined by TG)	(at 73°C)	2.20%
	(at 350°C)	0.61%
	total	2.81%

*- all percentages are based on the dry weight of the pfa.
**- taking into account that there is 1.34wt.% of gypsum present in the pfa.

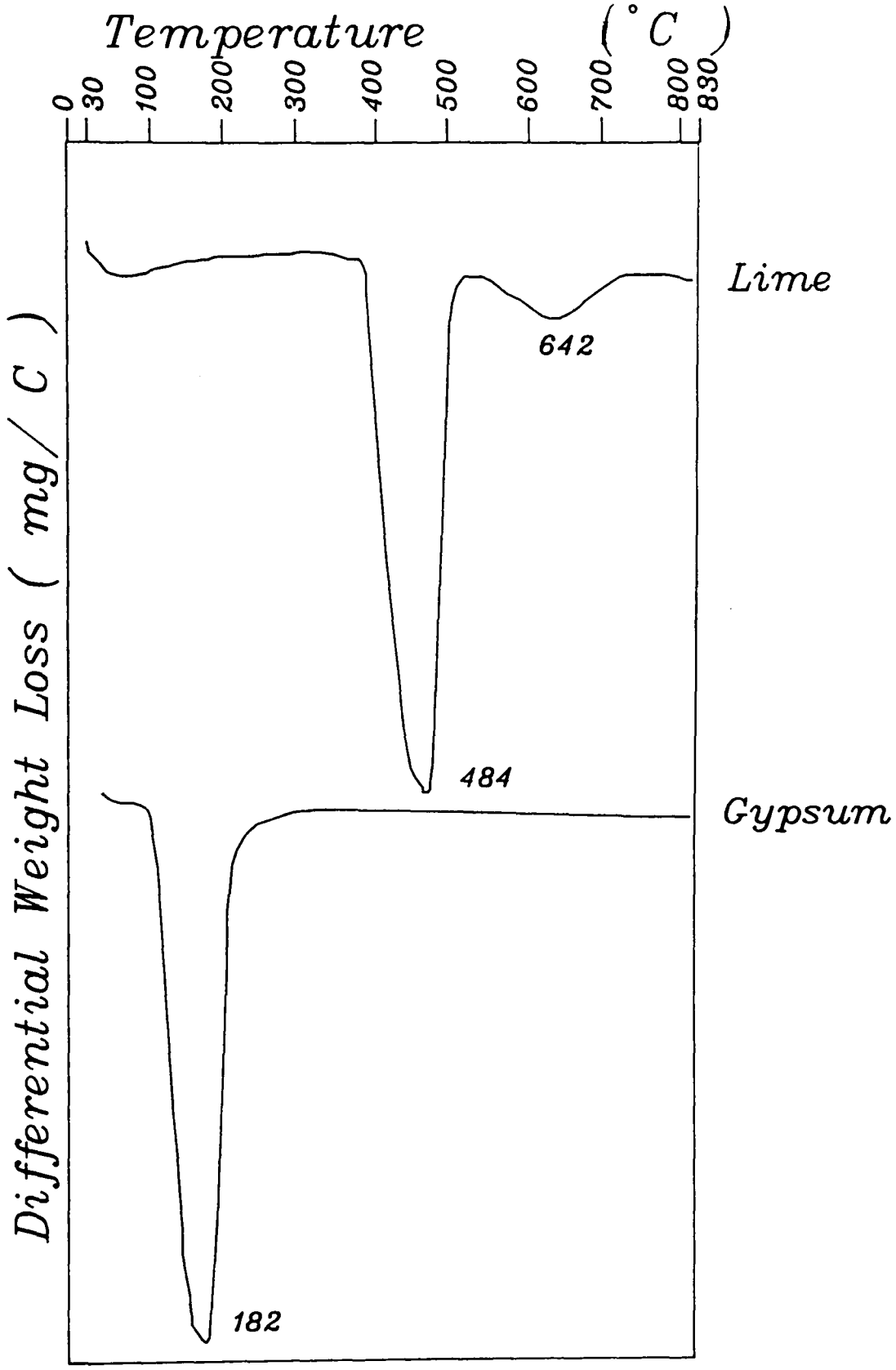


Figure 4.1 DTG thermograms of the lime and the gypsum.

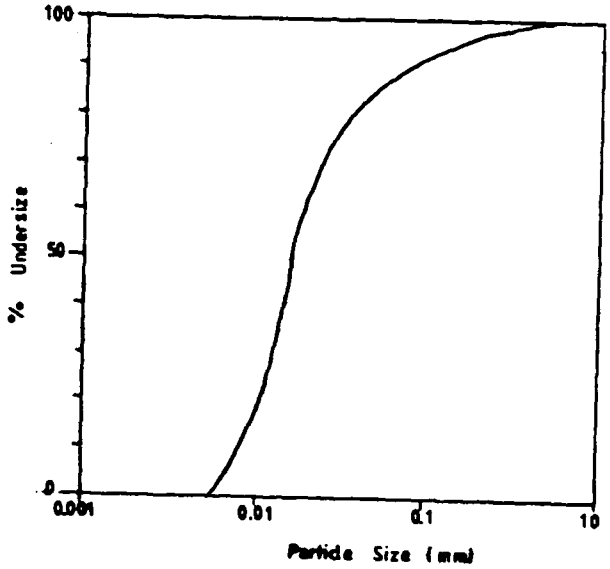


Figure 4.2 Grading curve for the pfa, determined from sieve analysis data and Coulter Counter data.

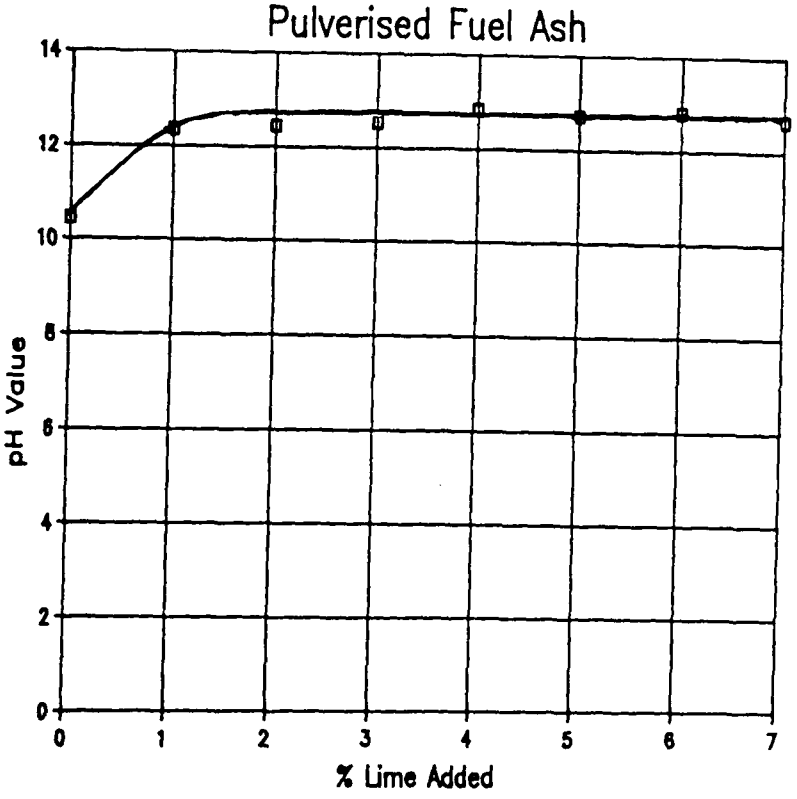


Figure 4.3 Graph of pH versus lime content for pfa-lime suspensions.

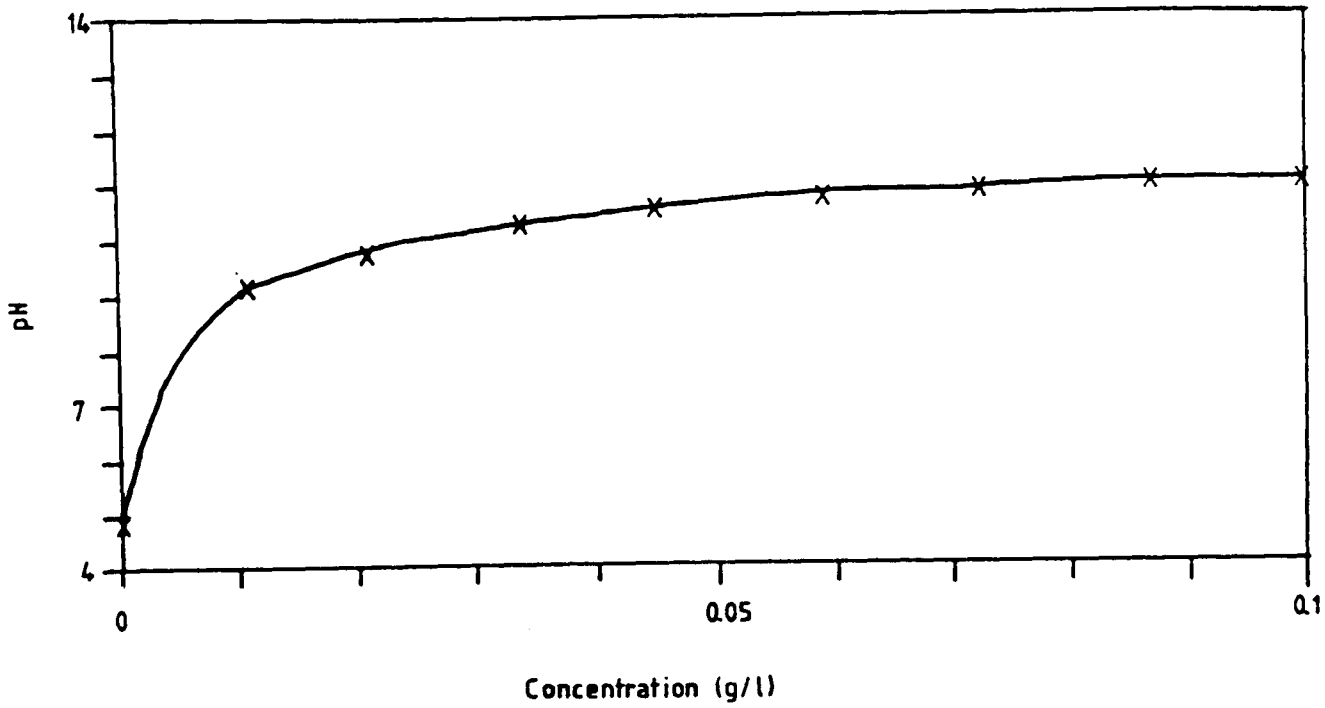


Figure 4.4 Graph of pH versus calcium hydroxide concentration for a saturated calcium sulphate solution.

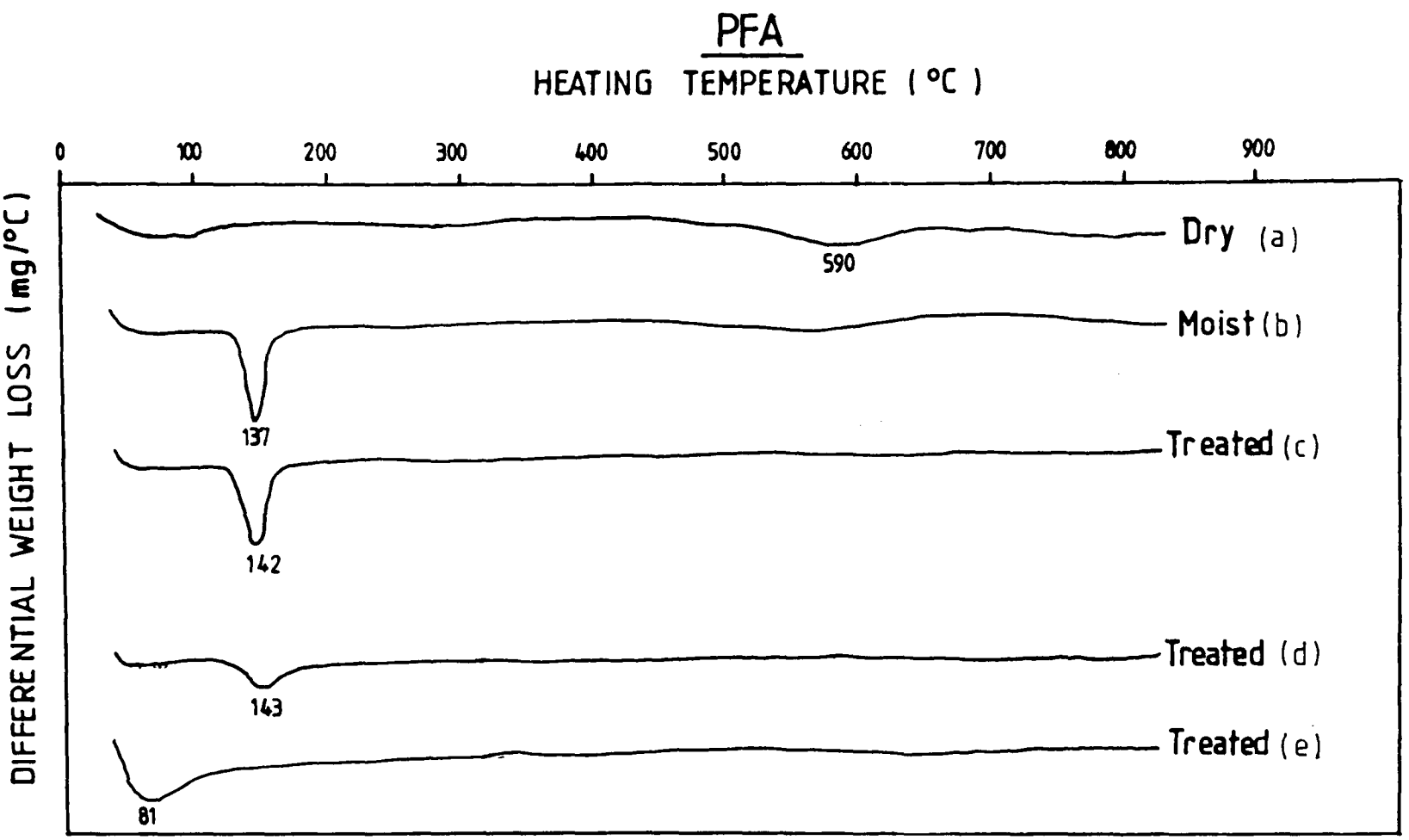


Figure 4.5 DTG thermograms of the pfa, a) in the dry state, b) moistened with 11wt.% of distilled water, c) after treatment with 4.68 x 10-2 molar HCl solution, d) after treatment with 9.36 x 10-2 molar HCl solution, and e) after treatment with 16.4 x 10-2 molar HCl solution.

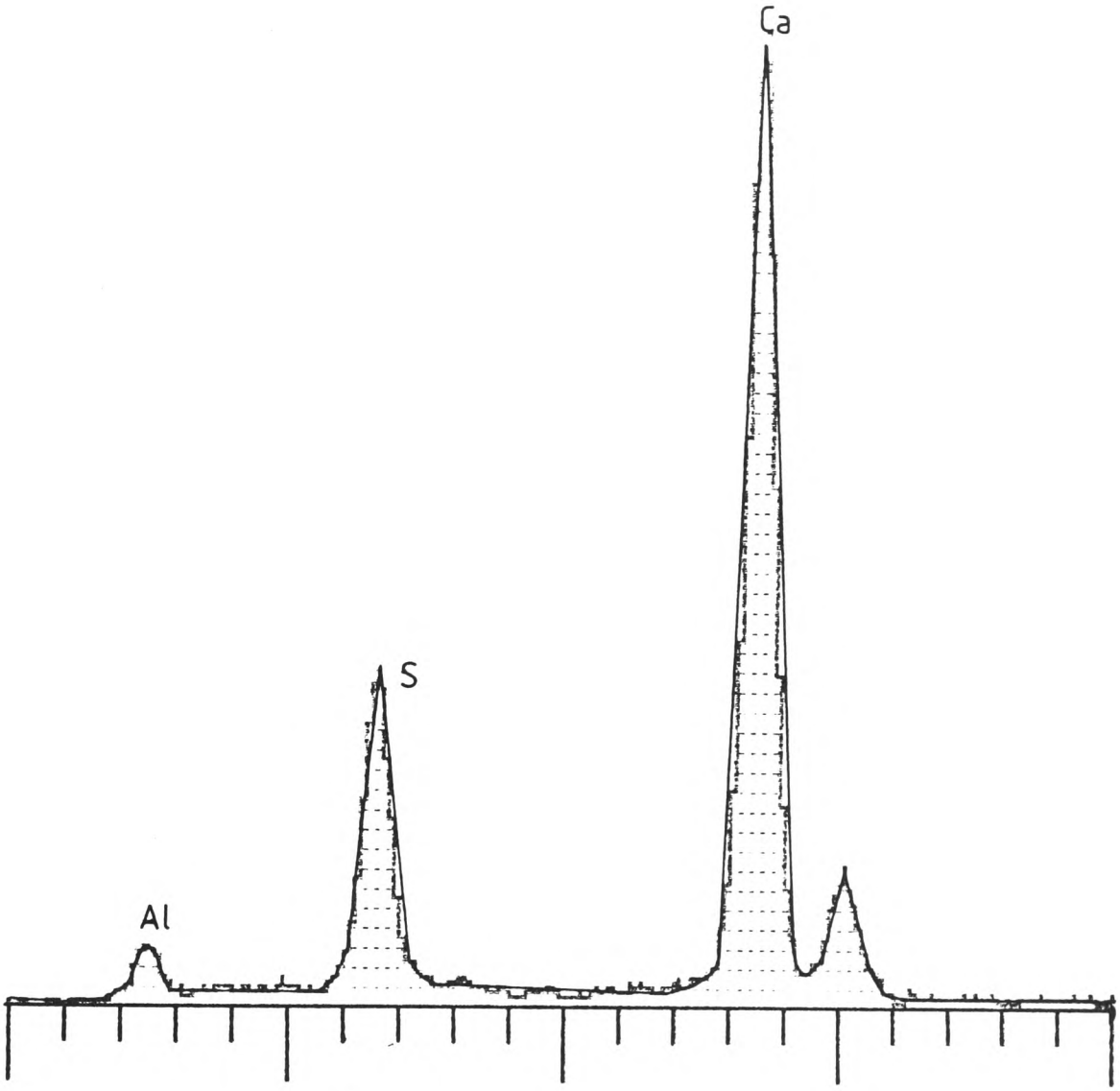


Figure 4.6 EDAX analysis of hexagonal ettringite rod.

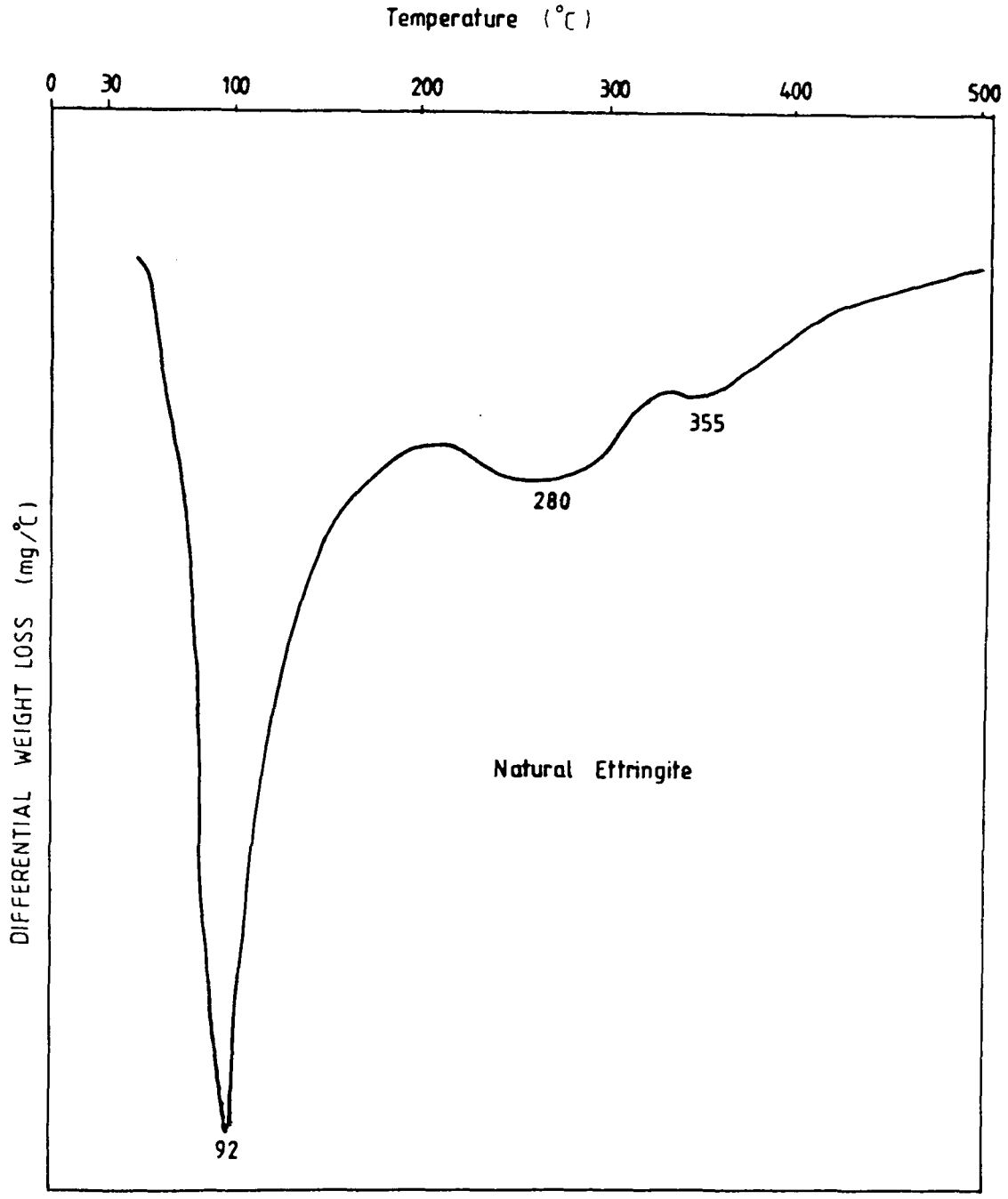


Figure 4.7 DTG thermogram of natural ettringite.

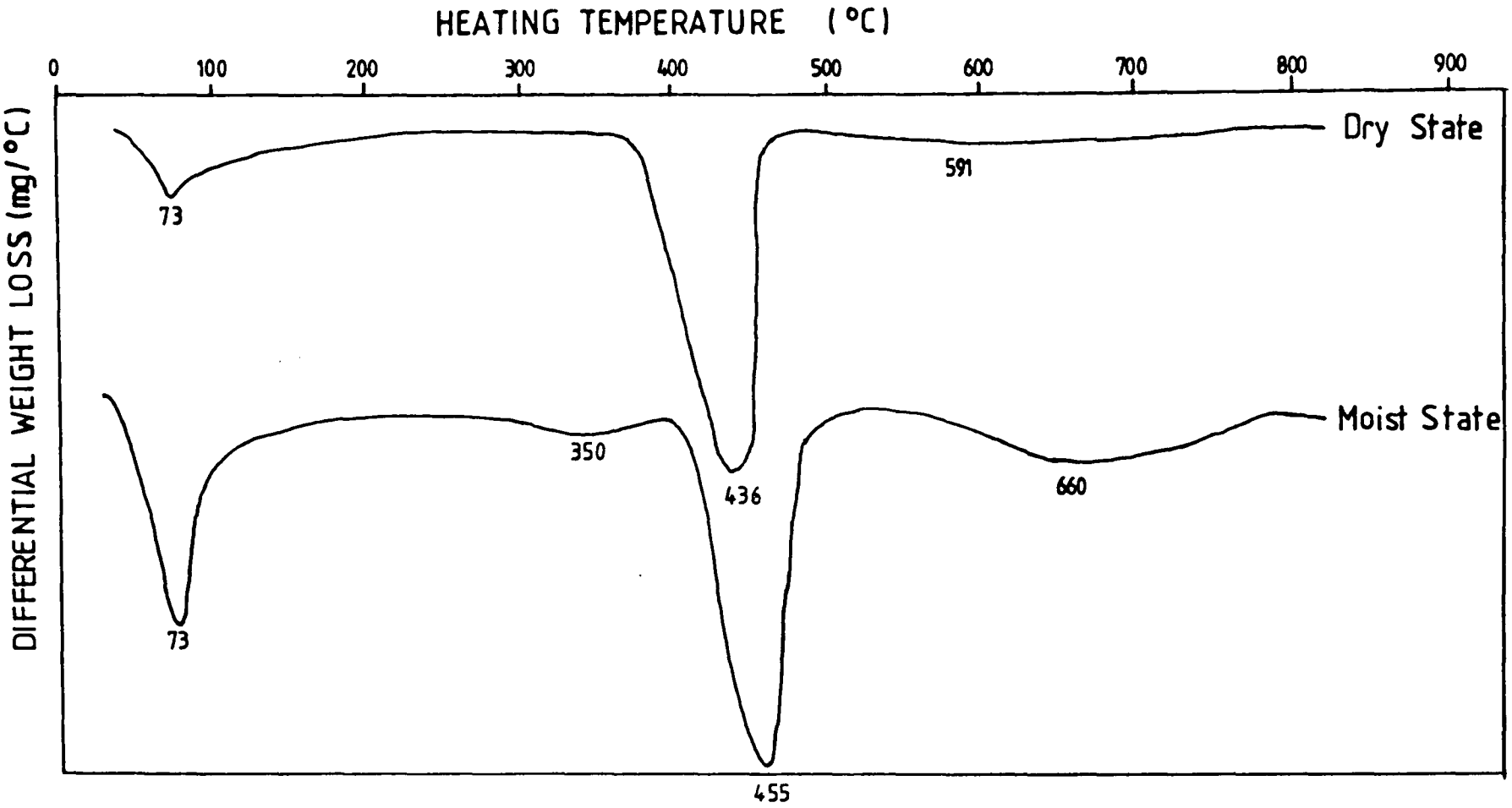


Figure 4.8 DTG thermograms for mixtures of pfa + 20wt.% added lime a) in the as supplied "dry" state and b) after being moistened with 30.2wt.% (of the weight of the pfa) of distilled water.

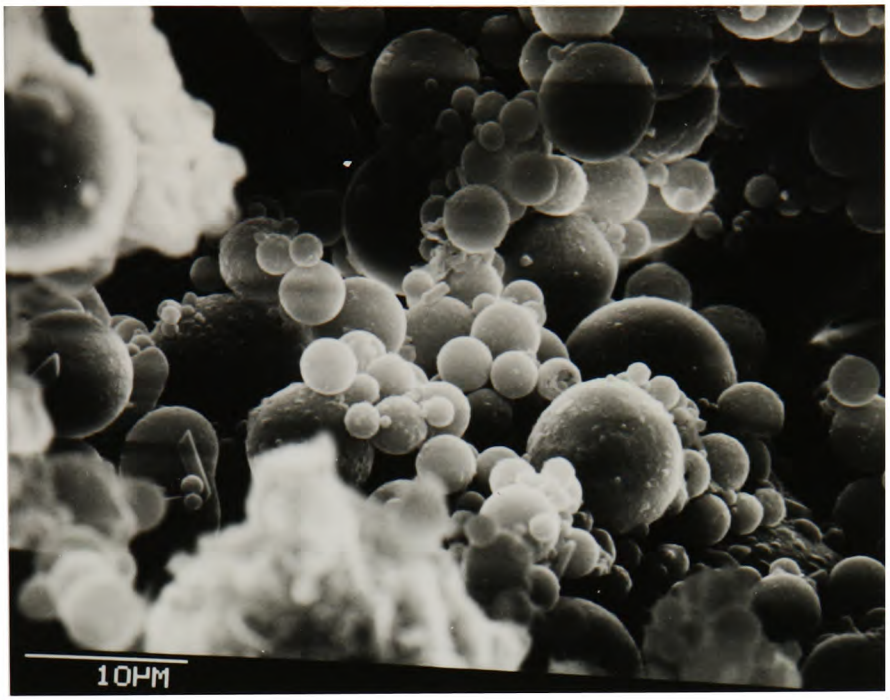


Plate 4.1 SEM micrograph showing typical pfa microstructure.

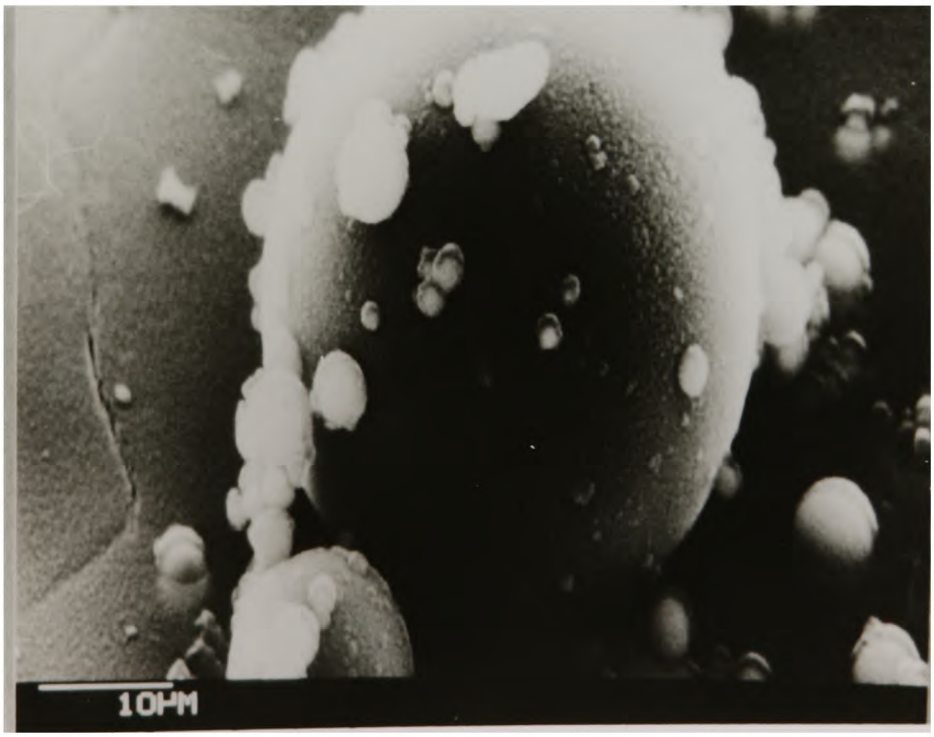


Plate 4.2 SEM micrograph showing typical "desulphurised" pfa particles.

Plate 4.3 SEM micrograph of natural ettringite.

Plate 4.4 SEM micrograph of hexagonal ettringite rods.

CHAPTER FIVE

TABLES, FIGURES AND PLATES

Table 5.1

Optimum moisture contents and maximum dry densities for pfa-lime mixtures.

% Lime Added	Optimum Moisture Content (% Water/Solid)	Maximum Dry Density* (g/cm^3)	
		$\underline{P}_m$	$\underline{P}_c$
0	18.0	1.58	1.66
5	23.8	1.44	1.51
10	24.9	1.40	1.48
15	25.7	1.40	1.47
20	25.2	1.37	1.48
25	25.3	1.40	1.47

* - Total Solids Dry Density.
$\underline{P}_m$ - Measured Values.
$\underline{P}_c$ - Calculated Values Assumining 0% Air Voids Present (see Appendix 3)

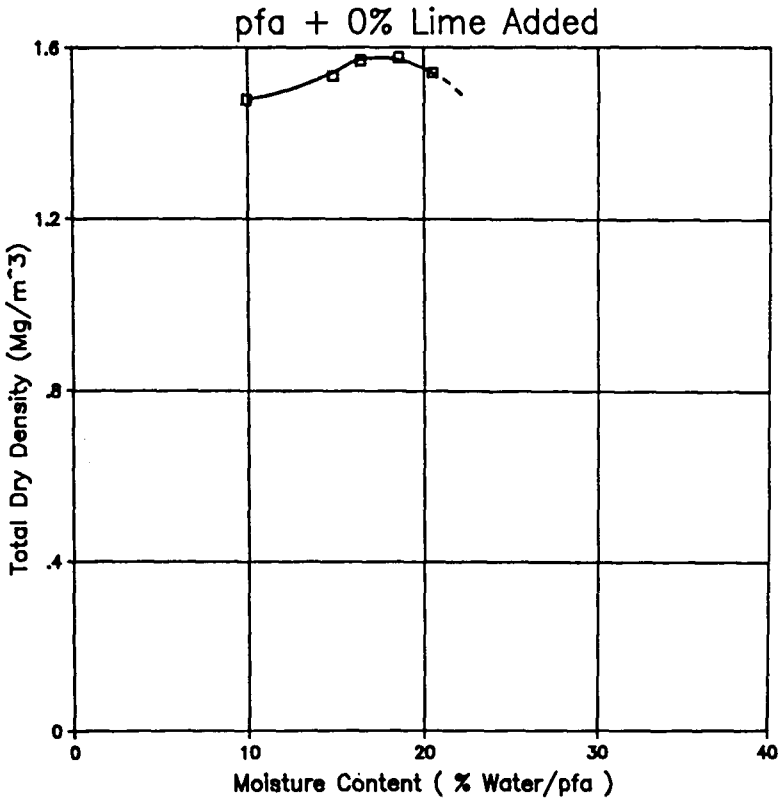


Figure 5.1 The compaction curve for pfa determined using the Proctor hammer method.

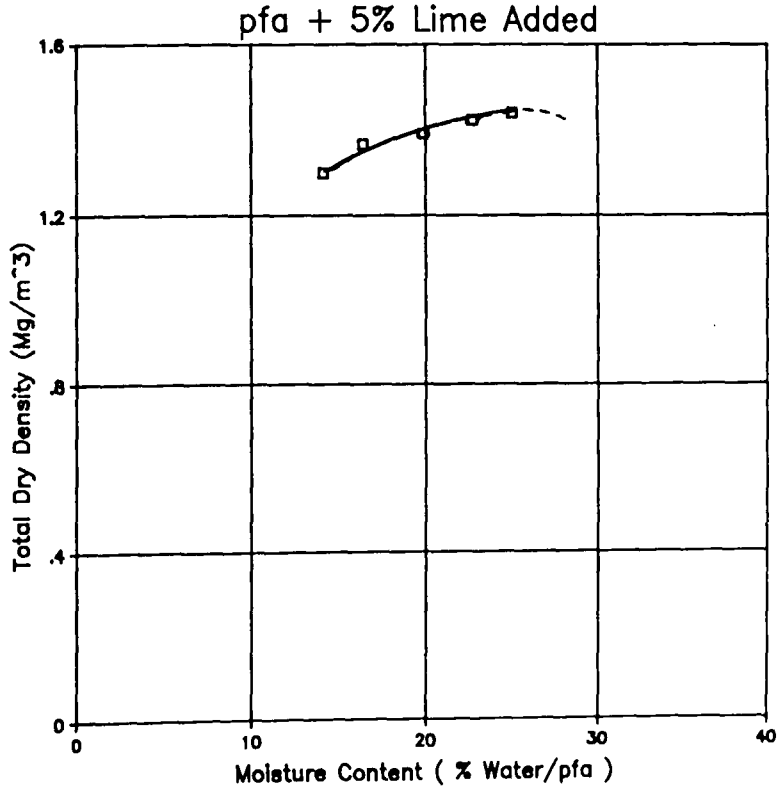


Figure 5.2 The compaction curve for pfa + 5wt.% lime mixtures determined using the Proctor hammer method.

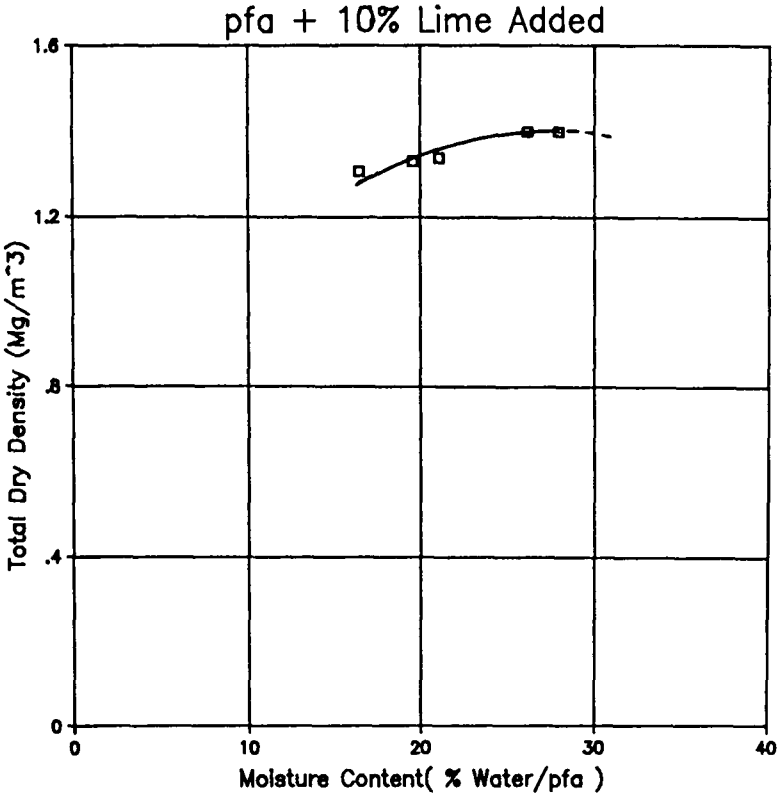


Figure 5.3 The compaction curve for pfa + 10wt.% lime mixtures determined using the Proctor hammer method.

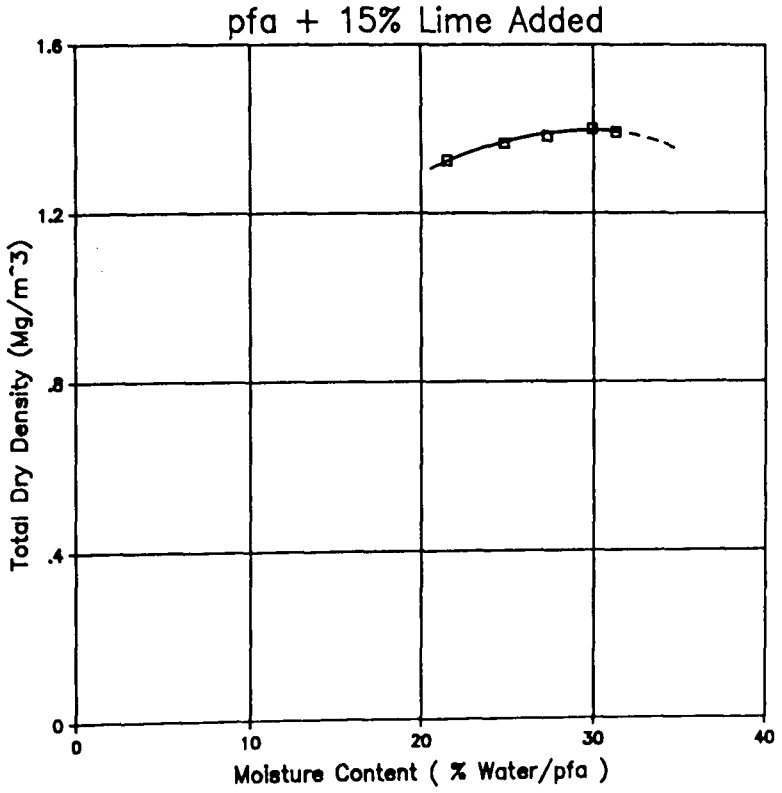


Figure 5.4 The compaction curve for pfa + 15wt.% lime mixtures determined using the Proctor hammer method.

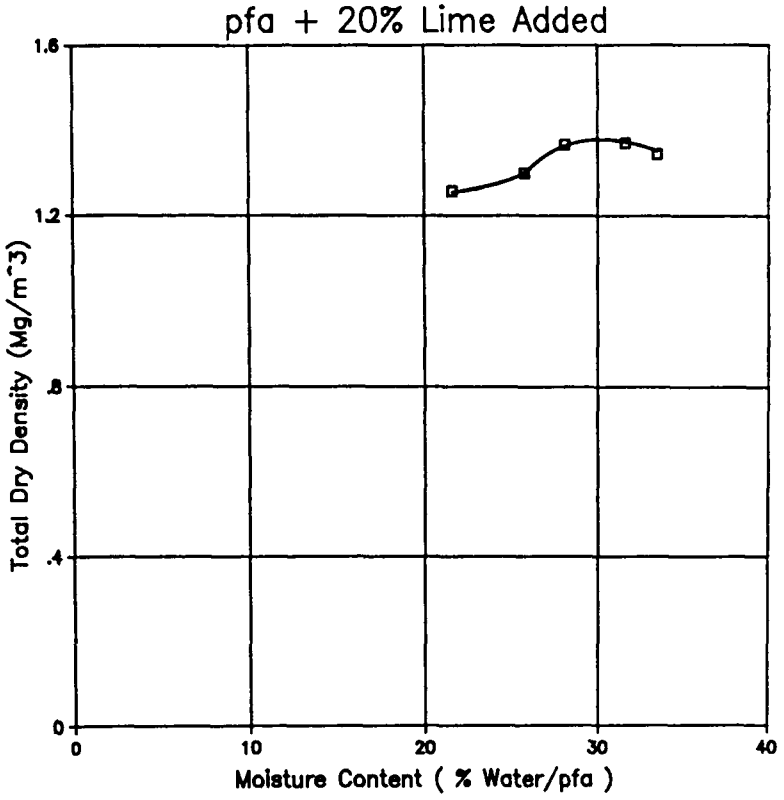


Figure 5.5 The compaction curve for pfa + 20wt.% lime mixtures determined using the Proctor hammer method.

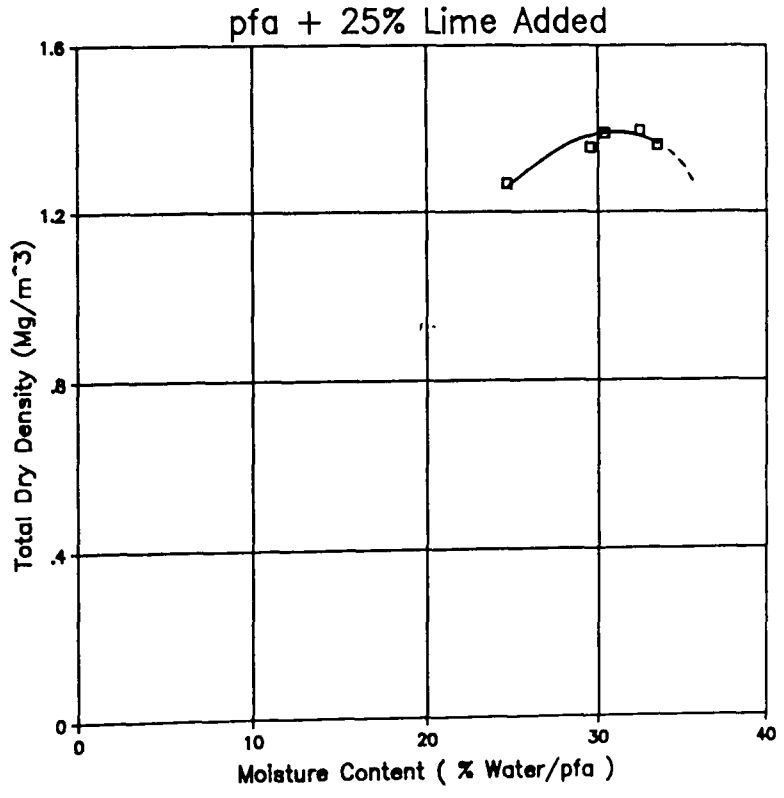


Figure 5.6 The compaction curve for pfa + 25wt.% lime mixtures determined using the Proctor hammer method.

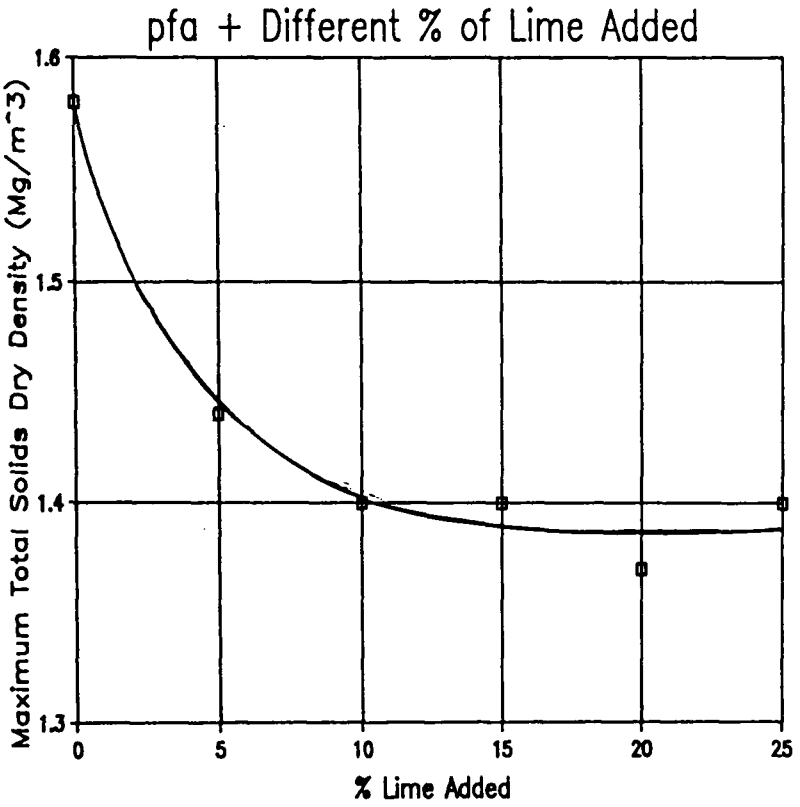


Figure 5.7 Maximum total solids dry density of pfa-lime mixtures versus wt.% of added lime.

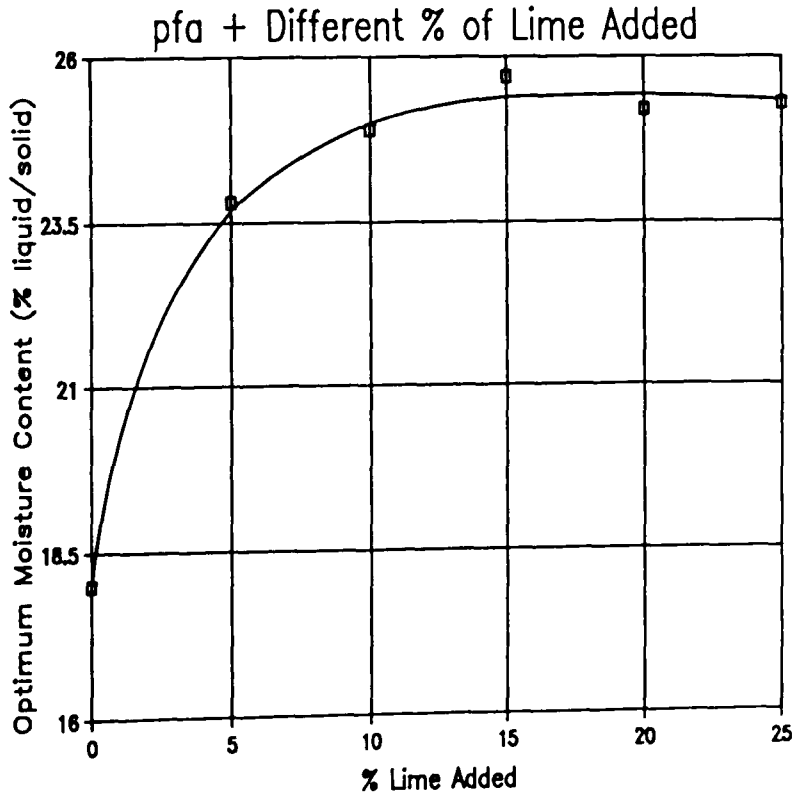


Figure 5.8 Optimum moisture content of pfa-lime mixtures versus wt.% of added lime.

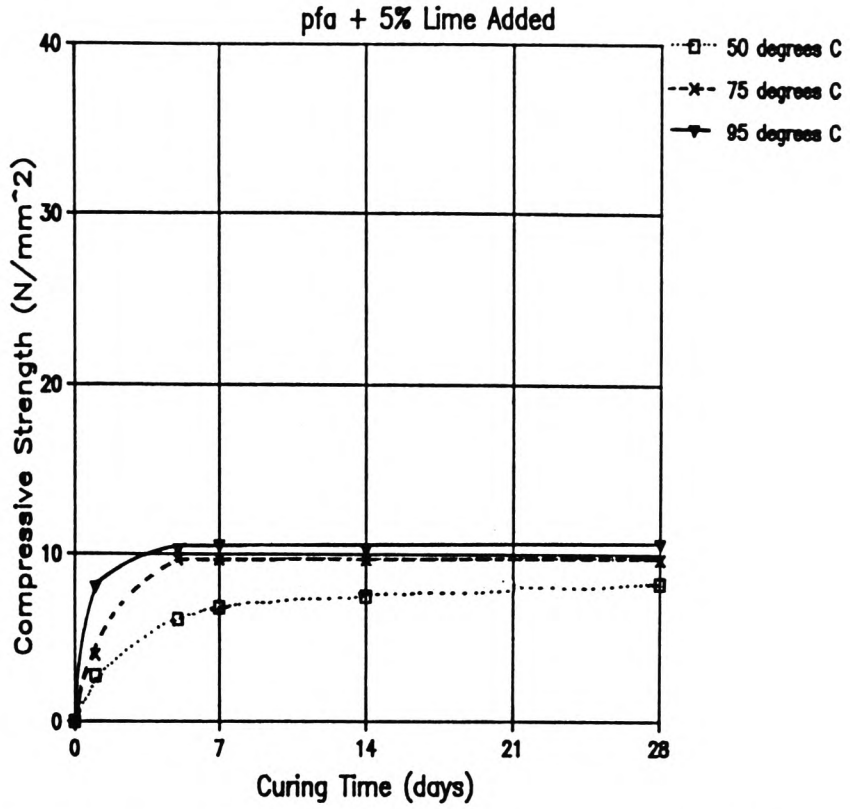


Figure 5.9 Compressive strength versus curing time for pfa + 5wt.% lime mixtures cured at 50°C, 75°C and 95°C.

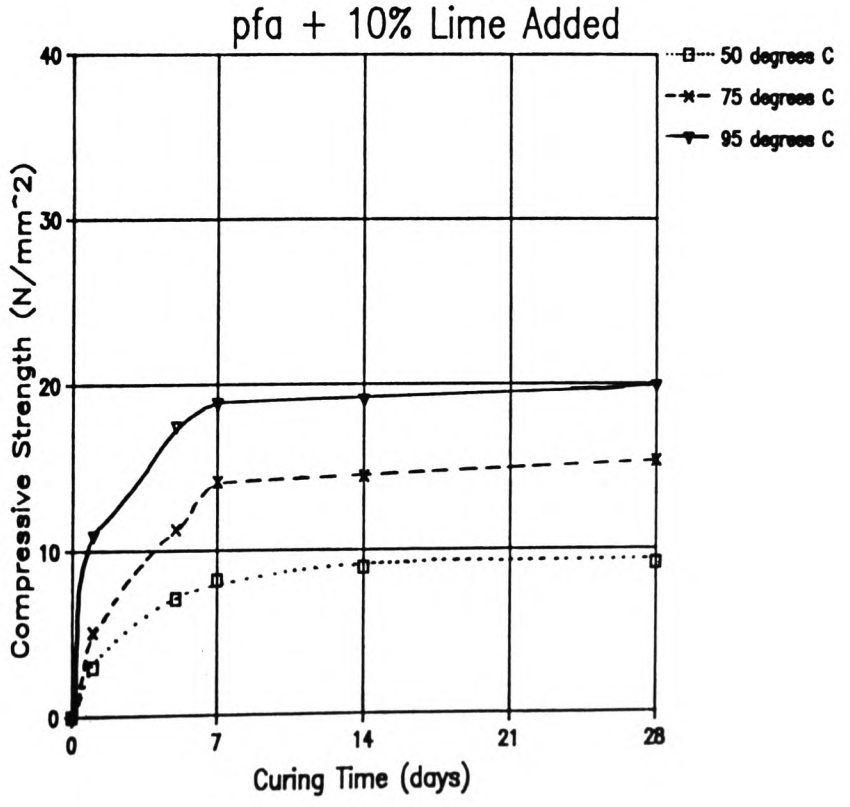


Figure 5.10 Compressive strength versus curing time for pfa + 10wt.% lime mixtures cured at 50°C, 75°C and 95°C.

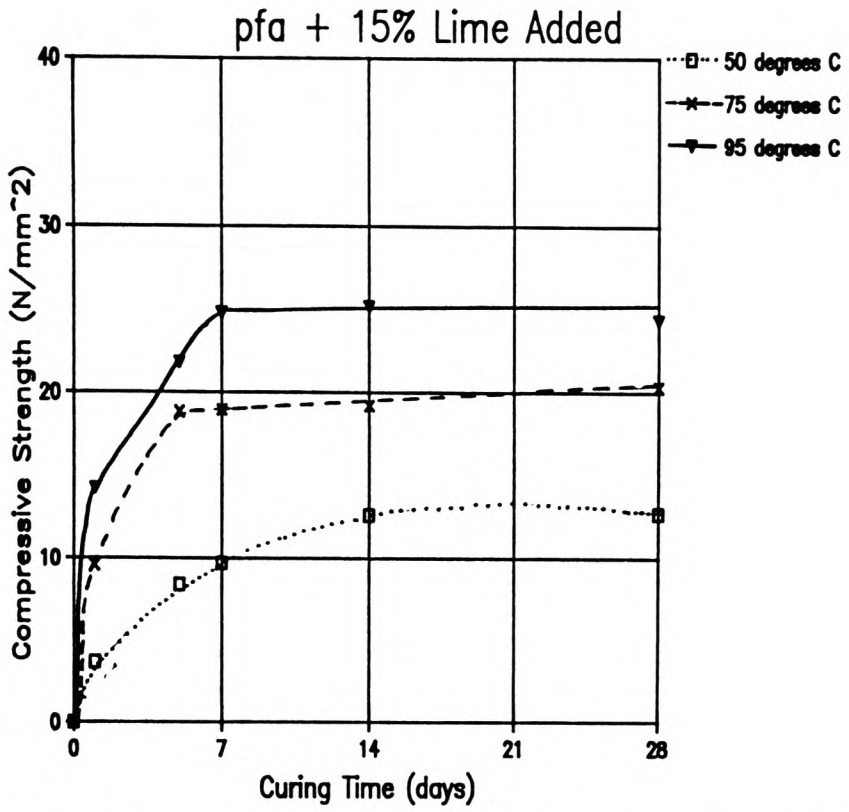


Figure 5.11 Compressive strength versus curing time for pfa + 15wt.% lime mixtures cured at 50°C, 75°C and 95°C.

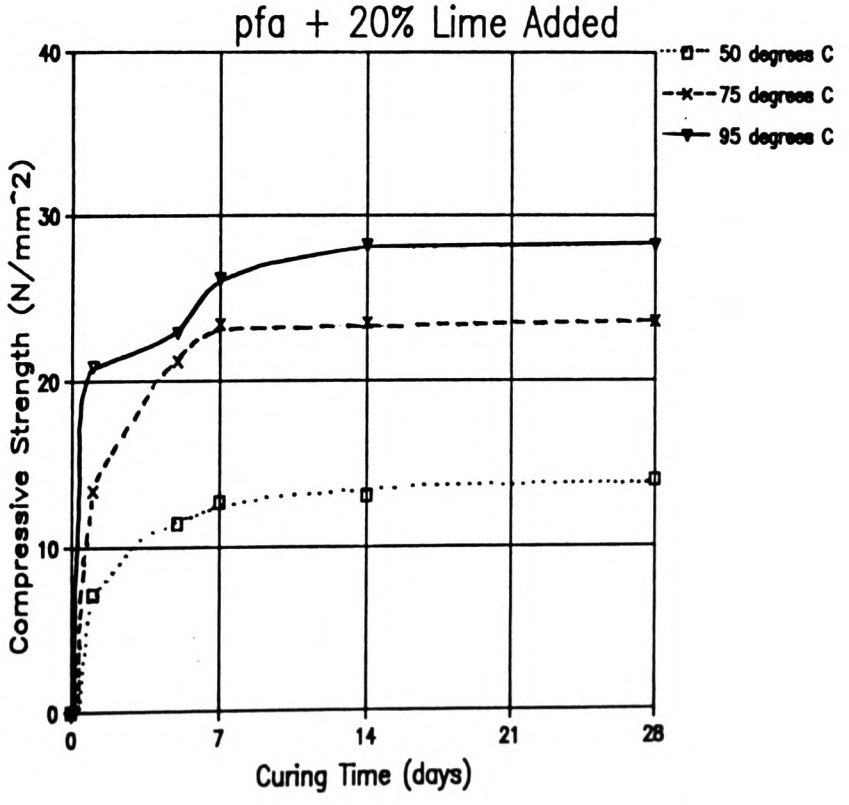


Figure 5.12 Compressive strength versus curing time for pfa + 20wt.% lime mixtures cured at 50°C, 75°C and 95°C.

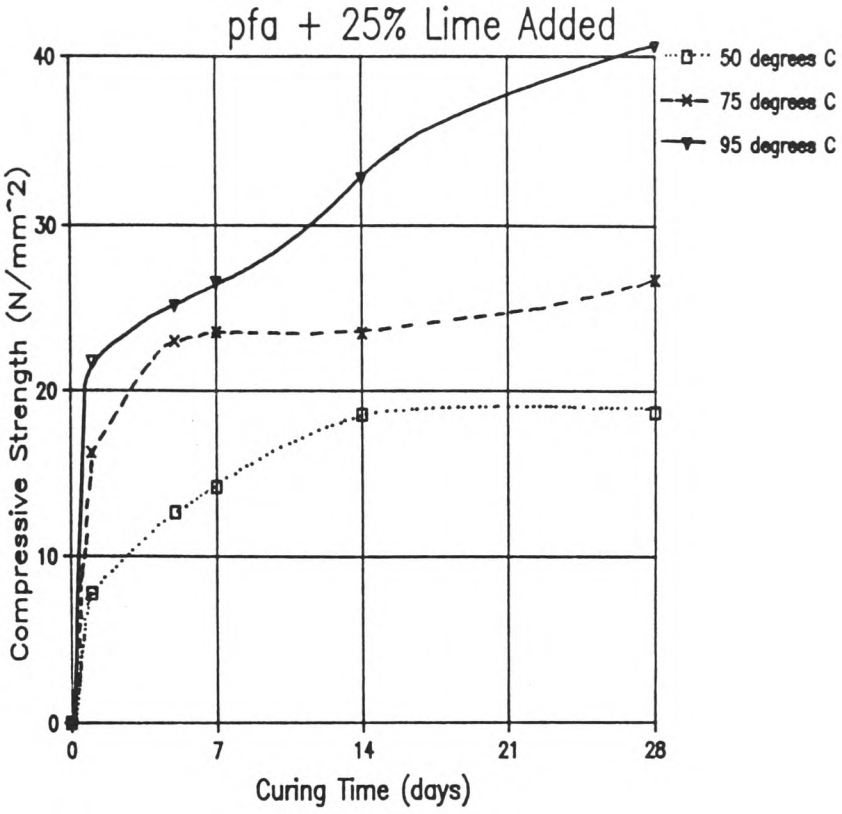


Figure 5.13 Compressive strength versus curing time for pfa + 25wt.% lime mixtures cured at 50°C, 75°C and 95°C.

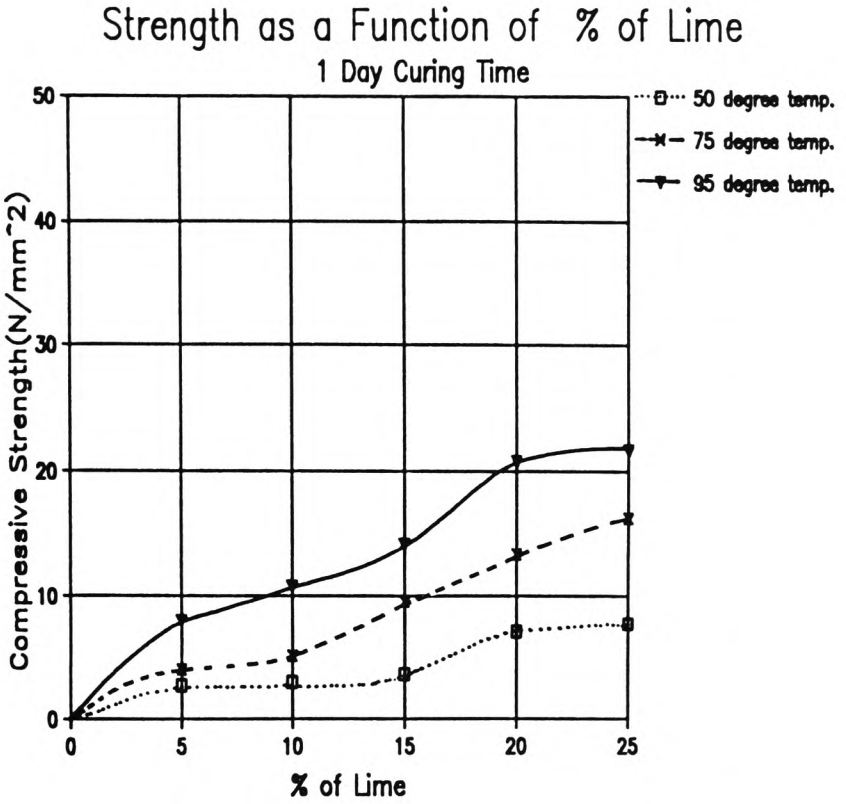


Figure 5.14 Compressive strength versus wt.% of added lime for pfa-lime mixtures cured for 1 day at 50°C, 75°C and 95°C.

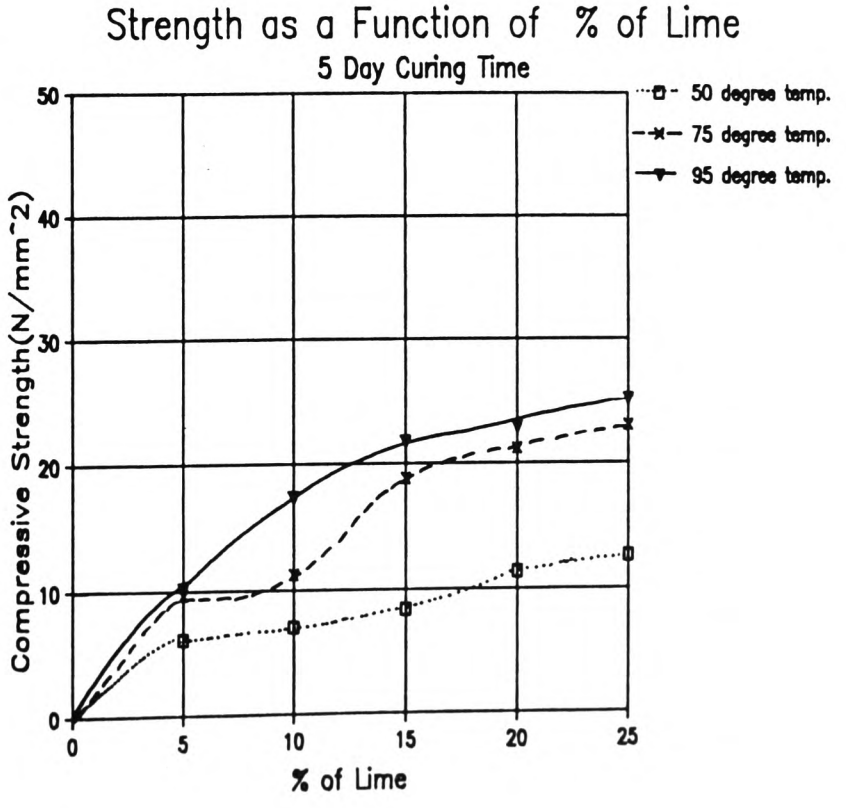


Figure 5.15 Compressive strength versus wt.% of added lime for pfa-lime mixtures cured for 5 day at 50°C, 75°C and 95°C.

CHAPTER SIX

TABLES, FIGURES AND PLATES

Table 6.1

Mass balance calculations derived from the DTG results for samples of pfa + 20wt.% lime cured at 50°C and 100% r.h. at curing times from 12 hours to 28 days.

A	B	C	D	E	F	G	H	I	J	K	L
12h	9.2794	.6979	.2590	8.2560	1.6512	.0949	.1641	.1214	.7369	8.9373	44.69
18h	9.4851	.6353	.1837	8.4250	1.6850	.0969	.0868	.0642	.8886	10.5396	52.70
24h	8.6646	.7563	.1701	7.6966	1.5393	.0885	.0815	.0603	.6341	8.2252	41.13
3d	8.5909	.4537	.3269	7.6586	1.5317	.0881	.2389	.1768	.8132	10.6133	53.07
7d	8.7535	.3936	.2384	7.7871	1.5574	.0896	.1488	.1101	.9642	12.3808	61.90
14d	8.2468	.3118	.2334	7.3379	1.4676	.0844	.1490	.1103	.9611	13.0846	65.42
28d	9.4368	.3485	.1815	8.3819	1.6764	.0964	.0851	.0630	1.1686	13.9415	69.71

Keys To Calculations,

A = Curing Time (h=hours,d=days)

B = Final Weight Of The Sample At ≈ 860°C

C = Weight of Free Lime in the Sample Detected by TG (mg)

D = Weight of $CaCO_3$ in the Sample Detected by TG (mg)

E = Weight of pfa in the Sample Calculated Using Equation 7 Of Appendix 1

F = Weight of the Lime in the Original Sample (mg = E*(20/100))

G = Weight of $CaCO_3$ in the Original Sample Calculated by Determining The Level Of The Carbonation In The Original Lime (mg)

H = Weight of $CaCO_3$ Formed During Curing (mg = D-G)

I = Weight of Lime Carbonated During Curing (mg = H*0.074)

J = Weight of Lime Consumed During Curing (mg = F-(C+G+I))

K = Percentage of Lime Consumed Based on pfa ((J/E)*100)

L = Percentage of Lime Consumed ((K/20)x100)

Table 6.2

Mass balance calculations derived from the DTG results for samples of pfa + 20wt.% lime cured at 75°C and 100% r.h. at curing times from 12 hours to 28 days.

A	B	C	D	E	F	G	H	I	J	K	L
12h	8.6202	.3251	.5165	7.7174	1.5435	.0888	.4277	.3165	.8131	10.5360	52.68
18h	9.2299	.4276	.2579	8.2120	1.6424	.0944	.1808	.1338	.9866	11.9992	60.00
24h	9.5060	.3776	.1907	8.4448	1.6890	.0971	.0936	.0692	1.1450	13.5610	67.80
3d	9.4968	.2711	.1493	8.4294	1.6859	.0969	.0524	.0387	1.2791	15.1751	75.88
7d	9.4952	.2286	.1524	8.4285	1.6857	.0969	.0555	.0411	1.3191	15.6287	78.14
14d	11.1813	.0000	.1883	9.9267	1.9853	.1142	.0741	.0548	1.8164	18.2981	91.49
28d	9.8909	.0000	.1608	8.7801	1.7560	.1010	.0598	.0443	1.6108	18.3450	91.72

Keys To Calculations,

A = Curing Time (h=hours,d=days)

B = Final Weight Of The Sample At ≈ 860°C

C = Weight of Free Lime in the Sample Detected by TG (mg)

D = Weight of $CaCO_3$ in the Sample Detected by TG (mg)

E = Weight of pfa in the Sample Calculated Using Equation 7 Of Appendix 1

F = Weight of the Lime in the Original Sample (mg = E*(20/100))

G = Weight of $CaCO_3$ in the Original Sample Calculated By Determining The Level Of The Carbonation In The Original Lime (mg)

H = Weight of $CaCO_3$ Formed During Curing (mg = D-G)

I = Weight of Lime Carbonated During Curing (mg = H*0.074)

J = Weight of Lime Consumed During Curing (mg = F-(C+G+I))

K = Percentage of Lime Consumed Based on pfa ((J/E)*100)

L = Percentage of Lime Consumed ((K/20)x100)

Table 6.3

Mass balance calculations derived from the DTG results for samples of pfa + 20wt.% lime cured at 95°C and 100% r.h. at curing times from 12 hours to 28 days.

A	B	C	D	E	F	G	H	I	J	K	L
6 h	9.9201	.6487	.1906	8.8112	1.7622	.1013	.0892	.0660	.9462	10.7416	53.71
12 h	9.3207	.2377	.1952	8.2815	1.6563	.0952	.1000	.0740	1.2494	15.0838	75.42
18 h	9.2849	.1525	.1349	8.2394	1.6479	.0948	.0401	.0297	1.3709	16.6448	83.22
24 h	9.8101	.1788	.1419	8.7053	1.7411	.1001	.0418	.0309	1.4312	16.4373	82.19
3 d	9.2834	.0000	.0851	8.2294	1.6459	.0946	.0000	.0000	1.5512	18.8500	94.25
7 d	10.8065	.0000	.0924	9.5784	1.9157	.1102	.0000	.0000	1.8055	18.8500	94.25
14 d	9.9957	.0000	.1511	8.8712	1.7742	.1022	.0491	.0363	1.6359	18.4480	92.24
28 d	9.6978	.0000	.0957	8.5980	1.7196	.0989	.0000	.0000	1.6207	18.8500	94.25

Keys To Calculations,

A = Curing Time (h=hours,d=days)

B = Final Weight of the Sample at ≈ 860°C (mg)

C = Weight of Free Lime in the Sample Detected by TG (mg)

D = Weight of CaCO3 in the Sample Detected by TG (mg)

E = Weight of pfa in the Sample Calculated Using Equation 7 of Appendix 1

F = Weight of the Lime in the Original Sample (mg = E*20/100)

G = Weight of CaCO3 in the Original Sample Calculated By Determining The Level Of The Carbonation In The Original Lime (mg)

H = Weight of CaCO3 Formed During Curing (mg = D-G)

I = Weight of Lime Carbonated During Curing (mg = H*0.074)

J = Weight of Lime Consumed In The Reaction (mg = F-{C+G+I})

K = Percentage of Lime Consumed Based on pfa ({J/E}*100)

L = Percentage of Lime Consumed ({K/20}*100)

Table 6.4

EDAX results from TEM studies of the fibrous gel formed in samples of pfa + 20wt.% lime cured at 75°C and 100% r.h. for the periods indicated.

Curing Time (days)	Mean Ca/Si	Mean Al/Si	Mean K/Si	Mean S/Si	Mean Na/Si	Mean Fe/Si	Number of Analyses
.5	Insufficient Fibrous Gel to Obtain a Representative Analyses						
.75	1.417	.272	.109	.061	.078	.107	13
1	1.369	.306	.184	.215	-	.062	6
3	1.444	.440	.093	.207	-	.280	4
7	1.183	.272	.105	.058	-	.055	7
14	1.061	.232	.071	.042	-	.040	12
28	1.027	.221	.115	.099	.050	.100	10
Average	1.225	.270	.108	.092	.064	.091	52
Ash Only	.049	.781	.112	.014	.158	.045	Calculated

Table 6.5

EDAX results from TEM studies of the fibrous gel formed in samples of pfa + 20wt.% lime cured at 95°C and 100% r.h. for the periods indicated.

Curing Time (days)	Mean Ca/Si	Mean Al/Si	Mean K/Si	Mean S/Si	Mean Na/Si	Mean Fe/Si	Number Analy
.5	Insufficient Fibrous Gel to Obtain a Representative Analyses						
.75	1.806	.367	.111	.035	.113	.104	8
1	1.581	.344	.126	.049	.108	.079	1(
3	1.287	.423	.135	.035	.182	.105	9
7	1.057	.397	.142	.029	.051	.247	1:
14	1.146	.402	.130	.030	.091	.116	1:
28	1.115	.425	.109	.043	.159	.081	9
Average	1.309	.393	.127	.036	.114	.126	5(
Ash Only	-	-	.112	.014	.158	.045	Clacu:

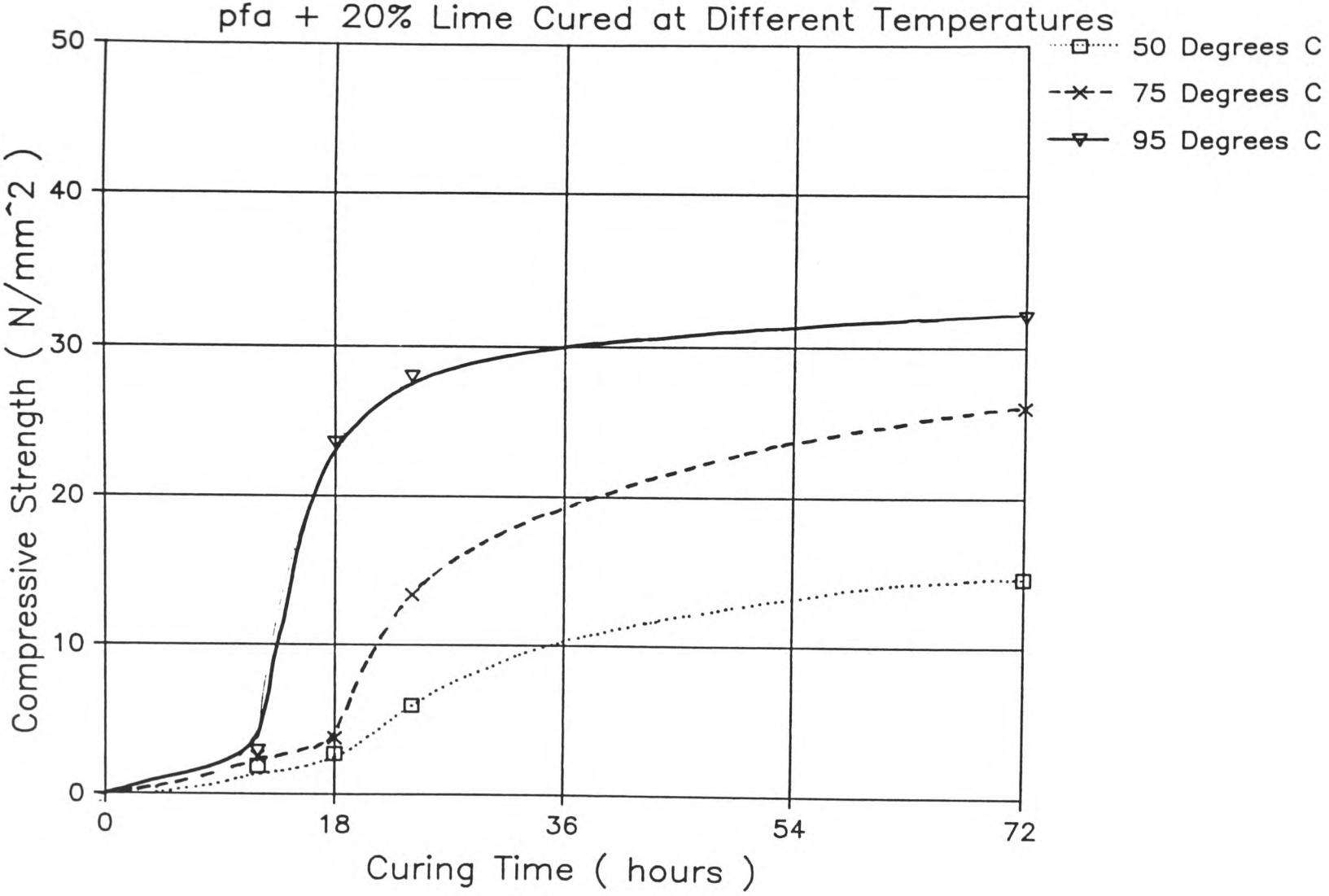


Figure 6.1 Compressive strength versus curing time (up to 3 days) for samples of pfa + 20wt.% lime cured at 50°C, 75°C and 95°C and at 100% r.h.

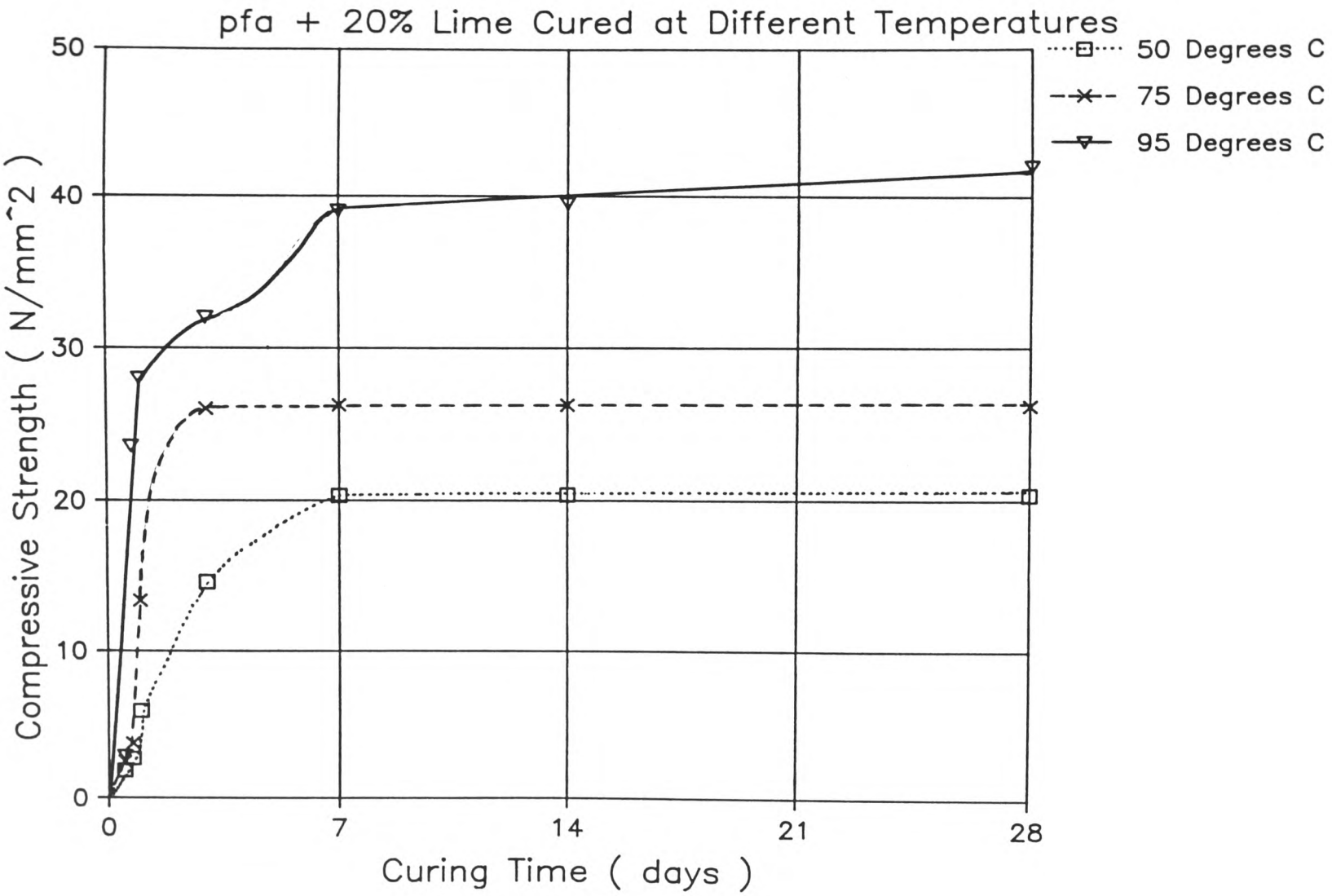


Figure 6.2 Compressive strength versus curing time (up to 28 days) for samples of pfa + 20wt.% lime cured at 50°C, 75°C and 95°C and at 100% r.h.

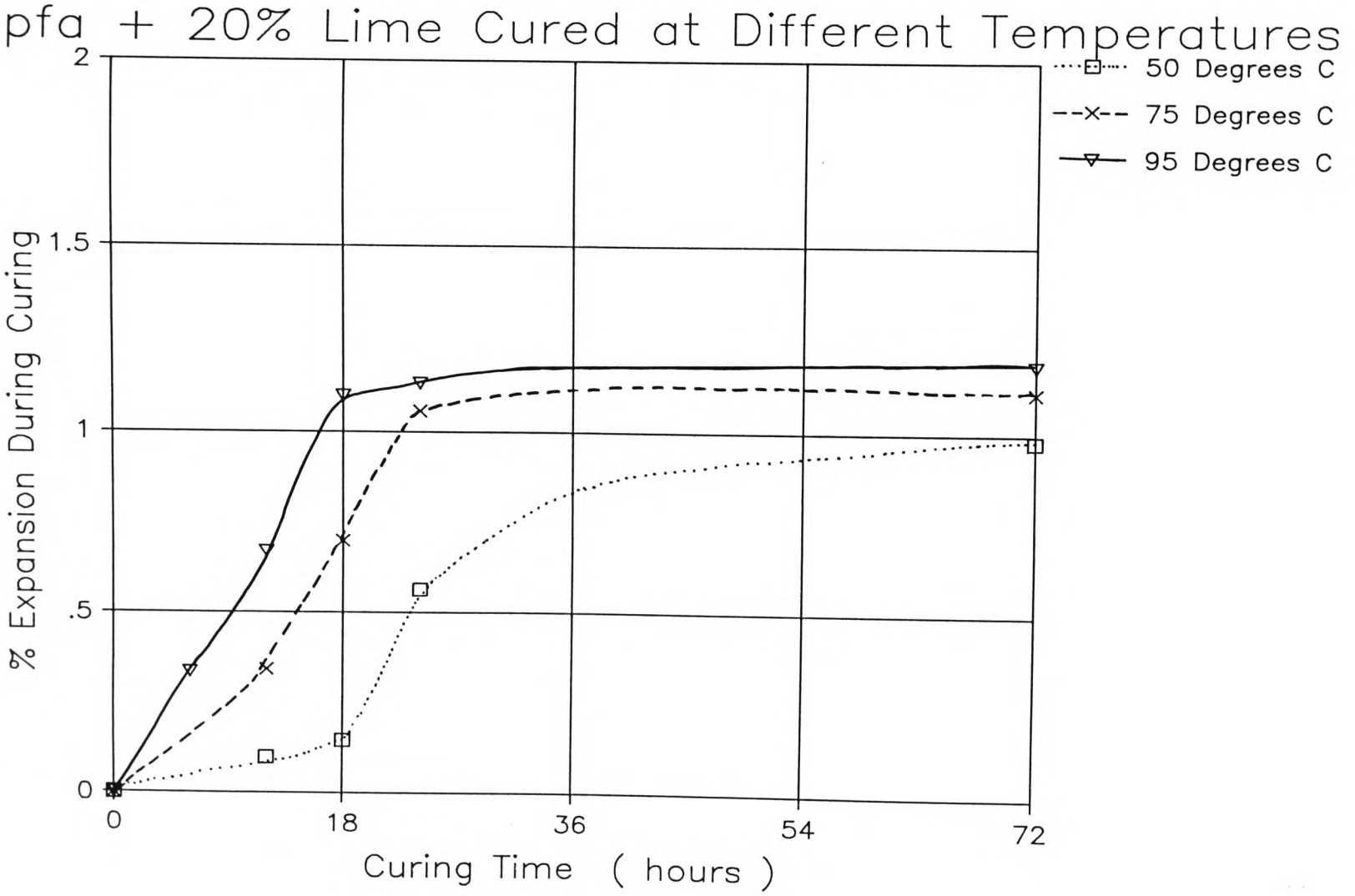


Figure 6.3 Expansion during curing versus curing time (up to 3 days) for samples of pfa + 20wt.% lime cured at 50°C, 75°C and 95°C and at 100% r.h.

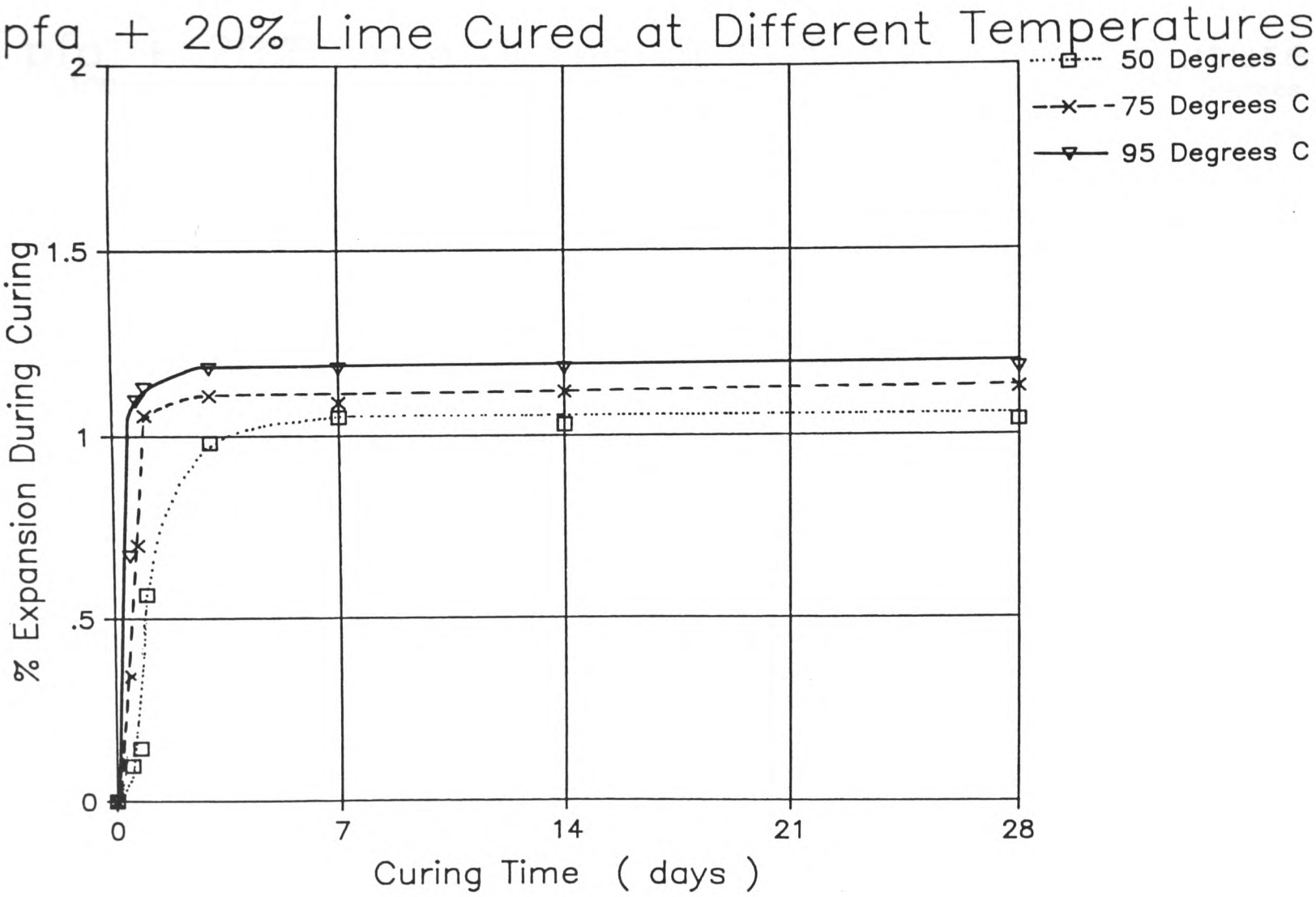


Figure 6.4 Expansion during curing versus curing time (up to 28 days) for samples of pfa + 20wt.% lime cured at 50°C, 75°C and 95°C and at 100% r.h.

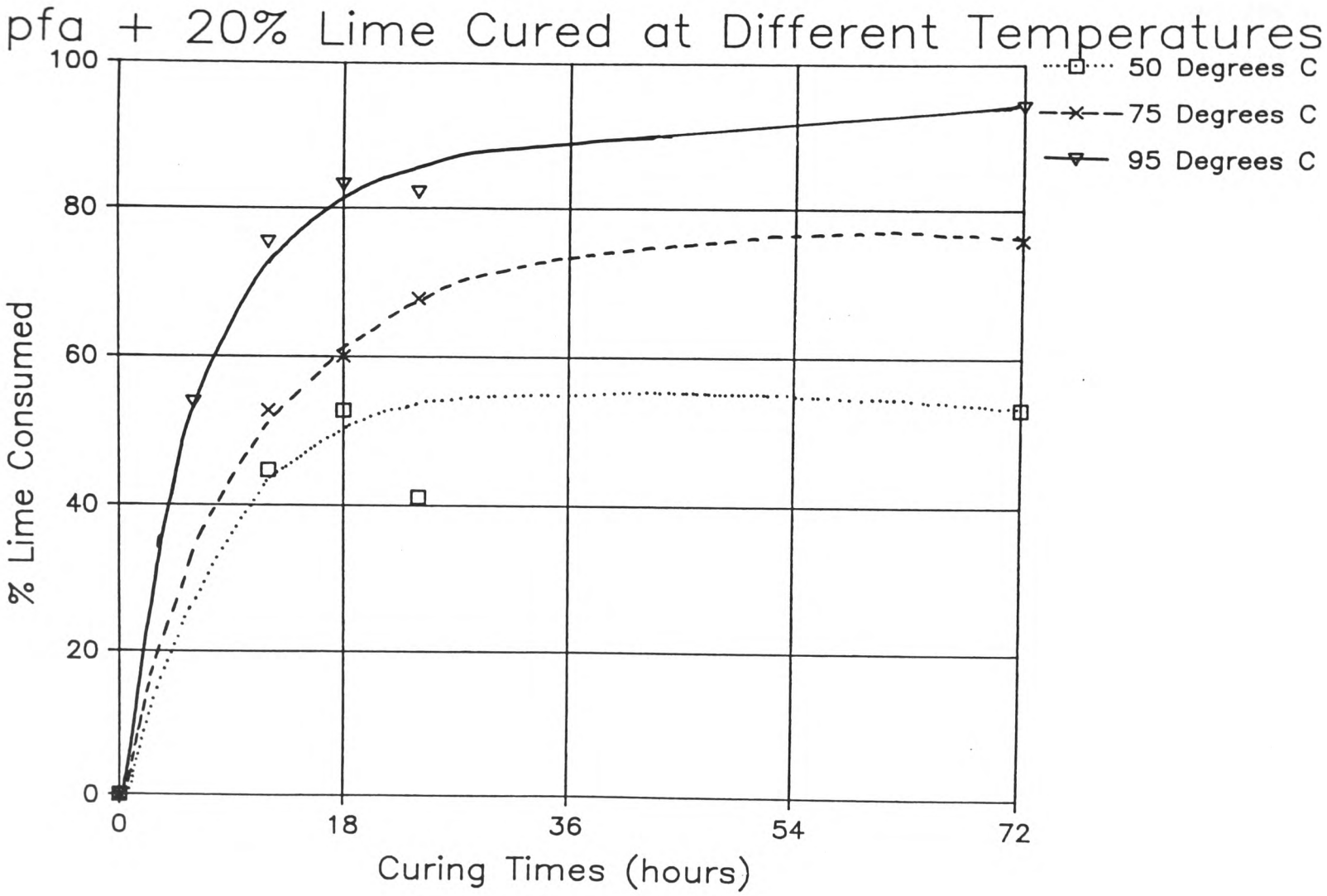


Figure 6.5 Percentage lime consumption (determined using DTG) versus curing time (up to 3 days) for samples of pfa + 20wt.% lime cured at 50°C, 75°C and 95°C and at 100% r.h.

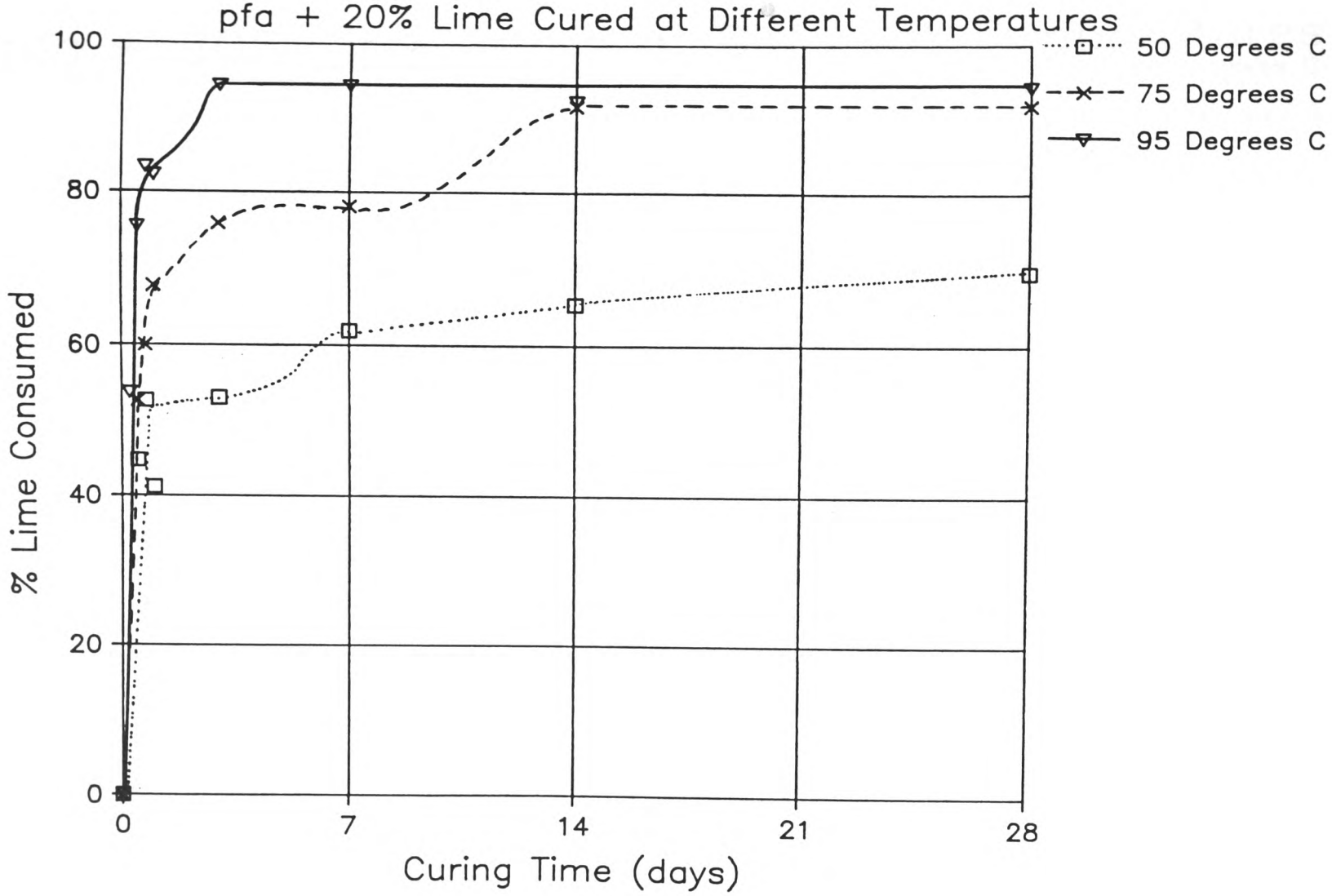


Figure 6.6 Percentage lime consumption (determined using DTG) versus curing time (up to 28 days) for samples of pfa + 20wt.% lime cured at 50°C, 75°C and 95°C and at 100% r.h.

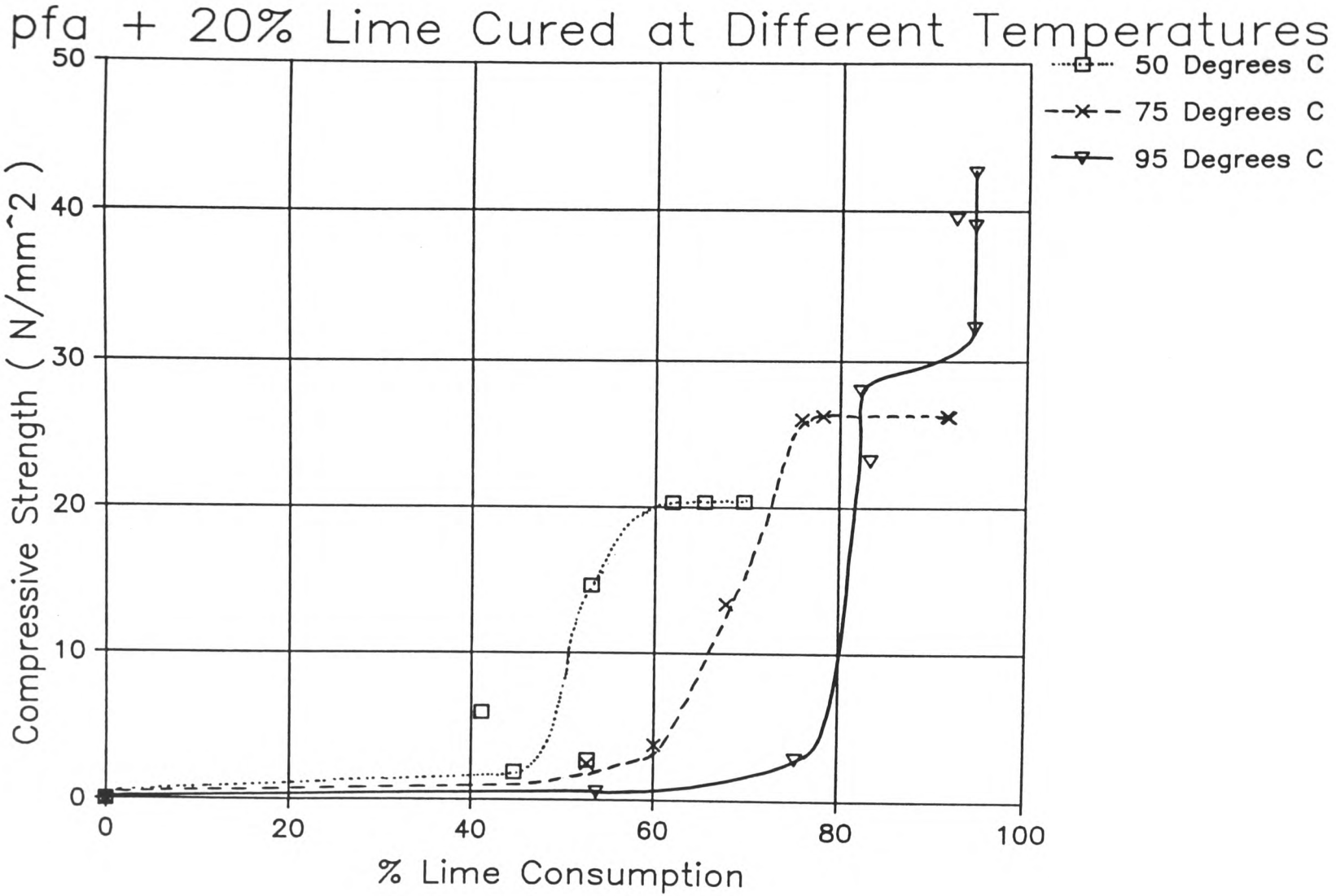


Figure 6.7 Compressive strength versus % lime consumption for samples of pfa + 20wt.% lime cured at 50°C, 75°C and 95°C and at 100 r.h. for up to 28 days.

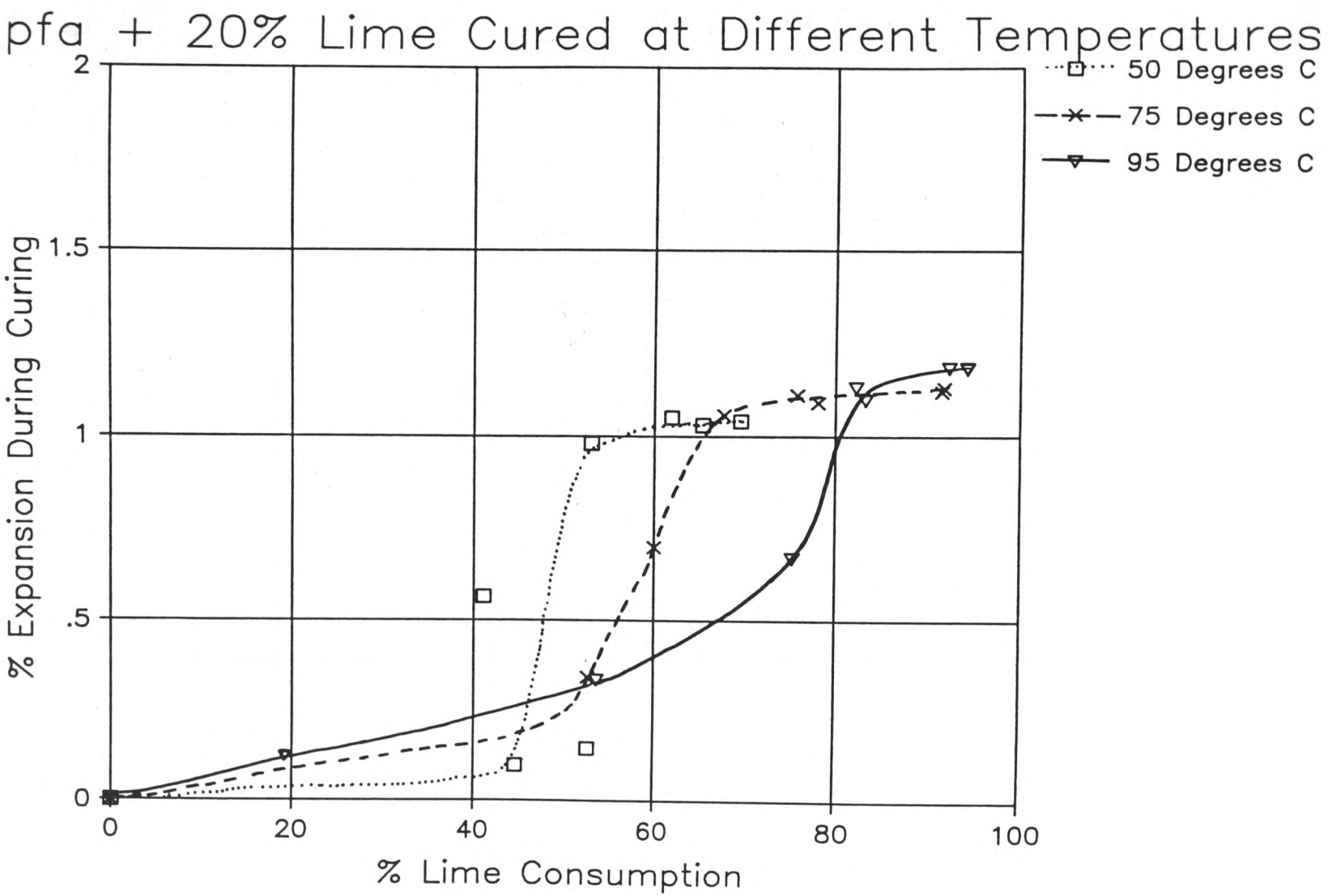


Figure 6.8 Expansion during curing versus % lime consumption for samples of pfa + 20wt.% lime cured at 50°C, 75°C and 95°C and at 100 r.h. for up to 28 days.

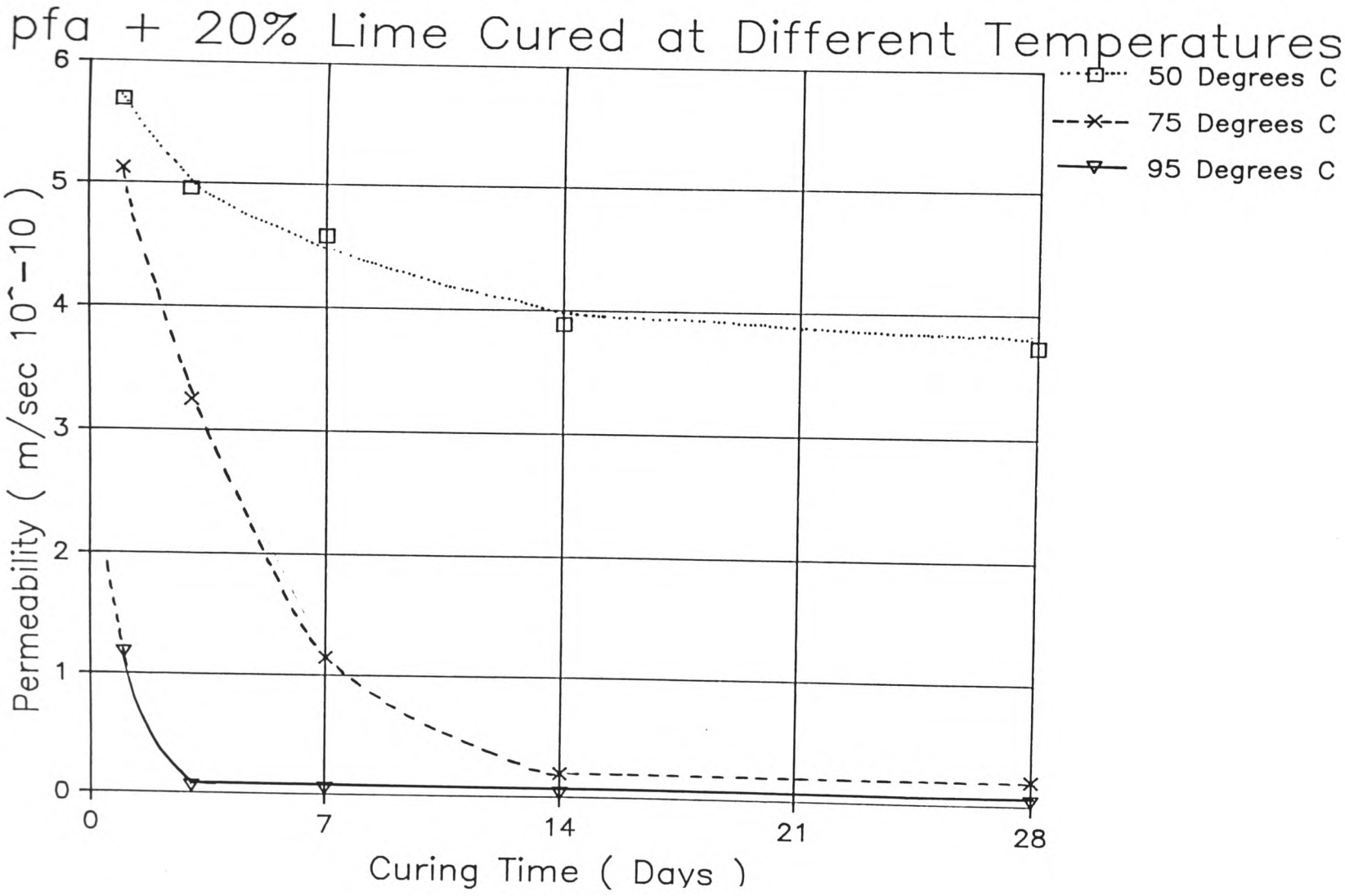


Figure 6.9 Permeability versus curing time (up to 28 days) for samples of pfa + 20wt.% lime cured at 50°C, 75°C and 95°C and at 100% r.h.

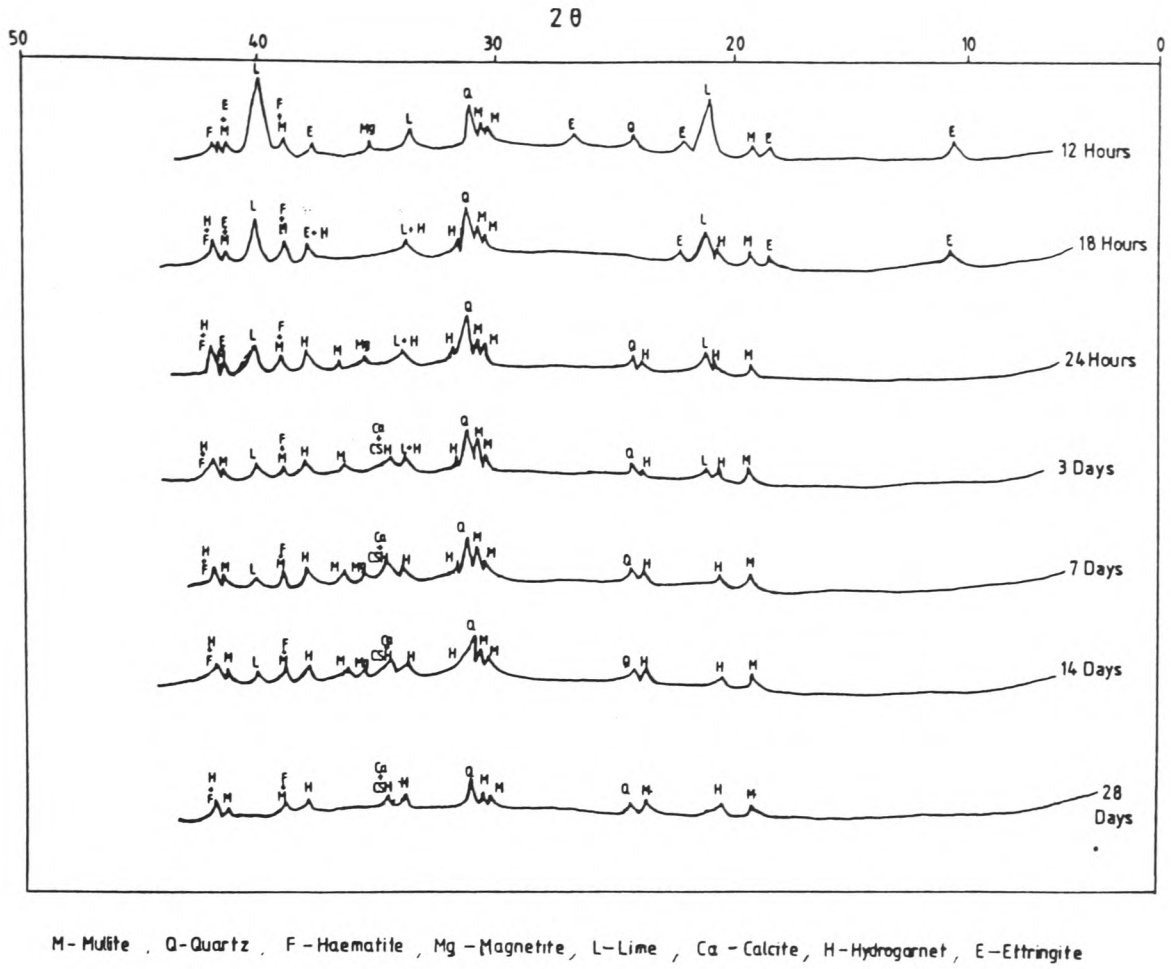
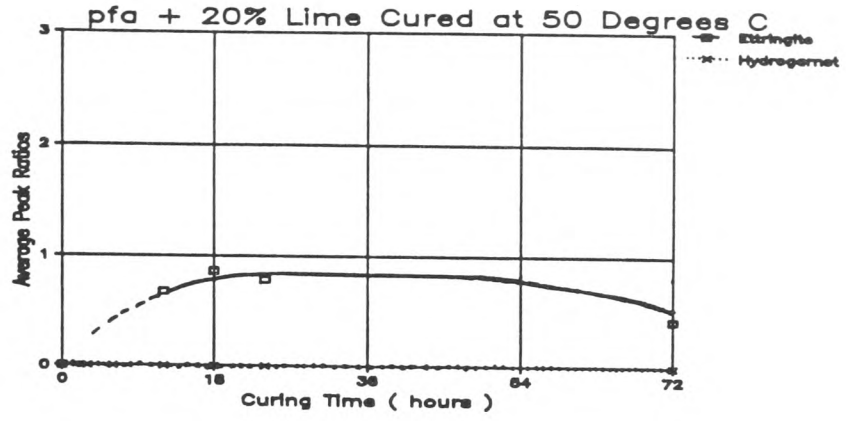
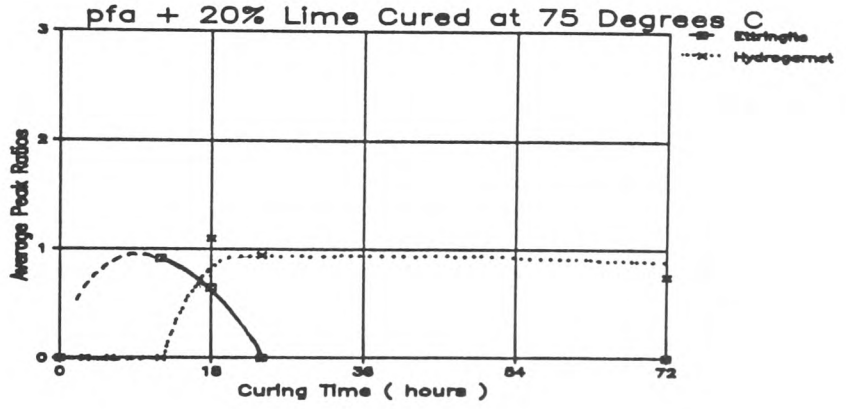


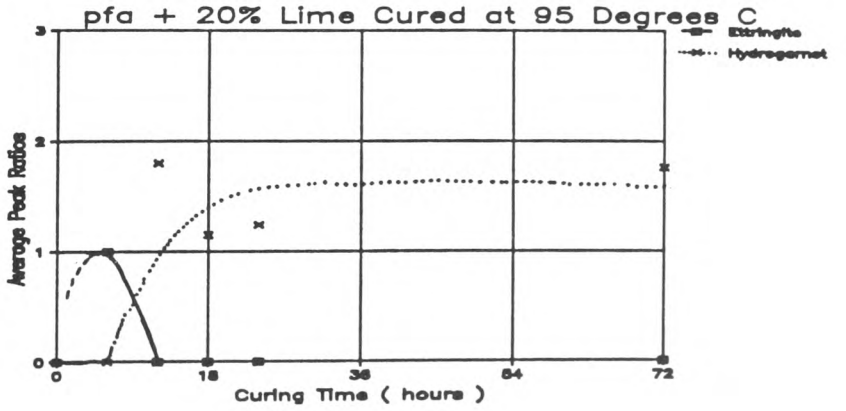
Figure 6.10 Typical XRD data. Samples in this case were of pfa + 20wt.% lime cured at 75°C and 100% r.h. from 12 hours to 28 days.



a



b



c

Diffraction peak ratios used were, ettringite d(0.561)/mullite d(0.539), hydrogarnet d(0.503)/mullite d(0.539) and hydrogarnet d(0.226)/mullite d(0.269).

Figure 6.11 Relative diffraction peak height ratios for ettringite and hydrogarnet plotted against curing time (up to 3 days) for samples of pfa + 20wt.% lime cured at a) 50°C, b) 75°C, and c) 95°C.

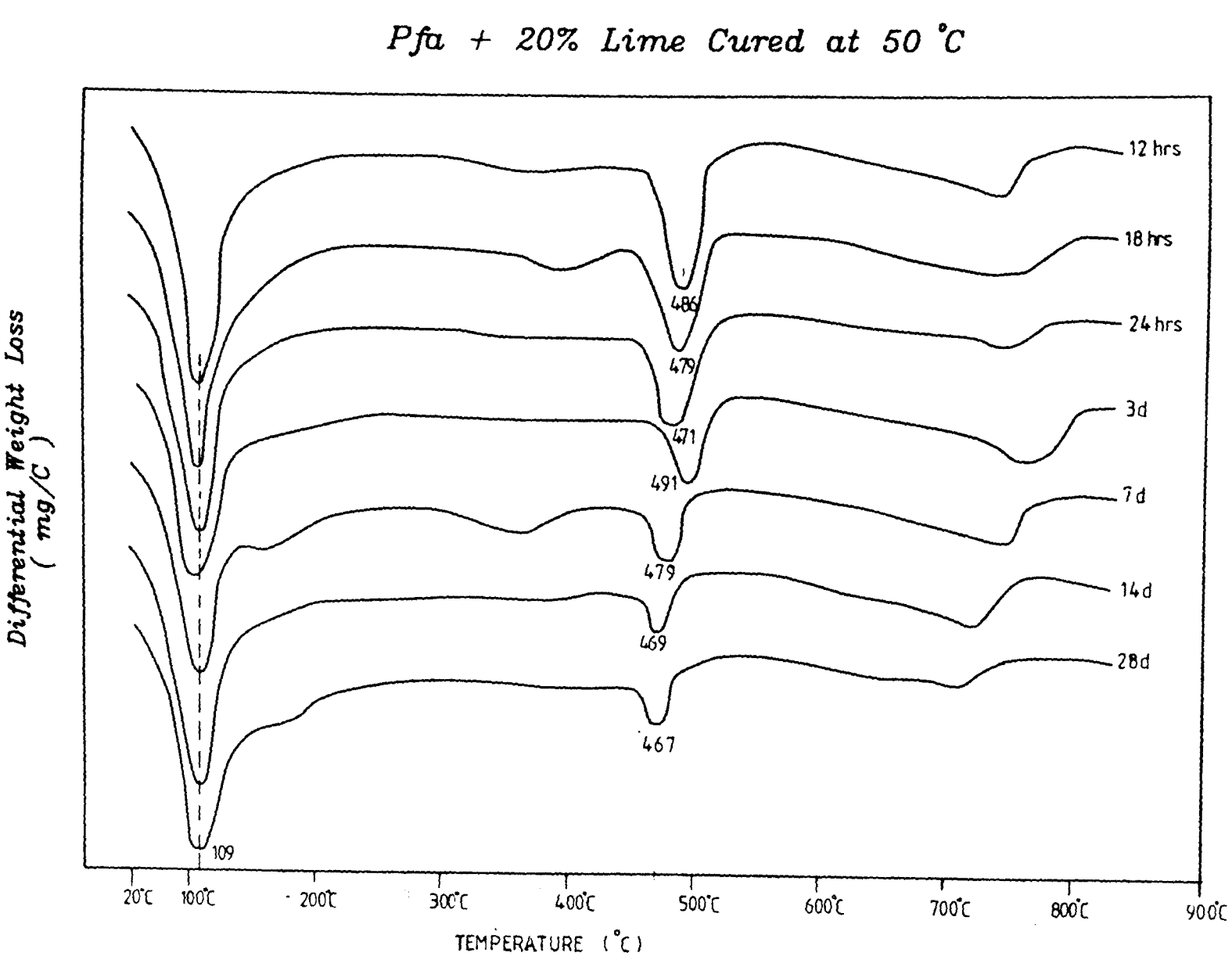


Figure 6.12 DTG thermograms for samples of pfa + 20wt.% lime cured at 50°C and 100% r.h. at curing times from 12 hours to 28 hours.

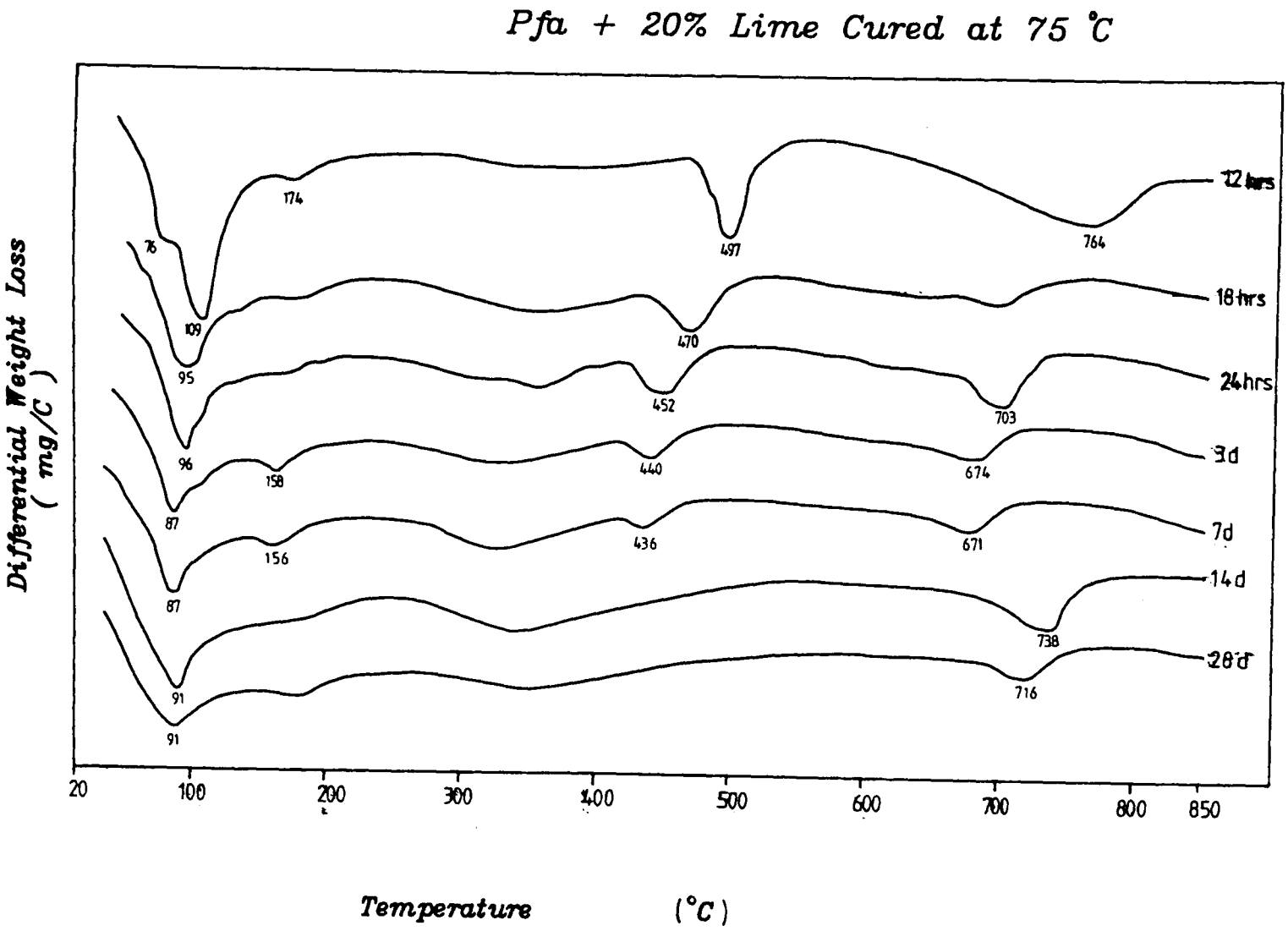


Figure 6.13 DTG thermograms for samples of pfa + 20wt.% lime cured at 75°C and 100% r.h. at curing times from 12 hours to 28 hours.

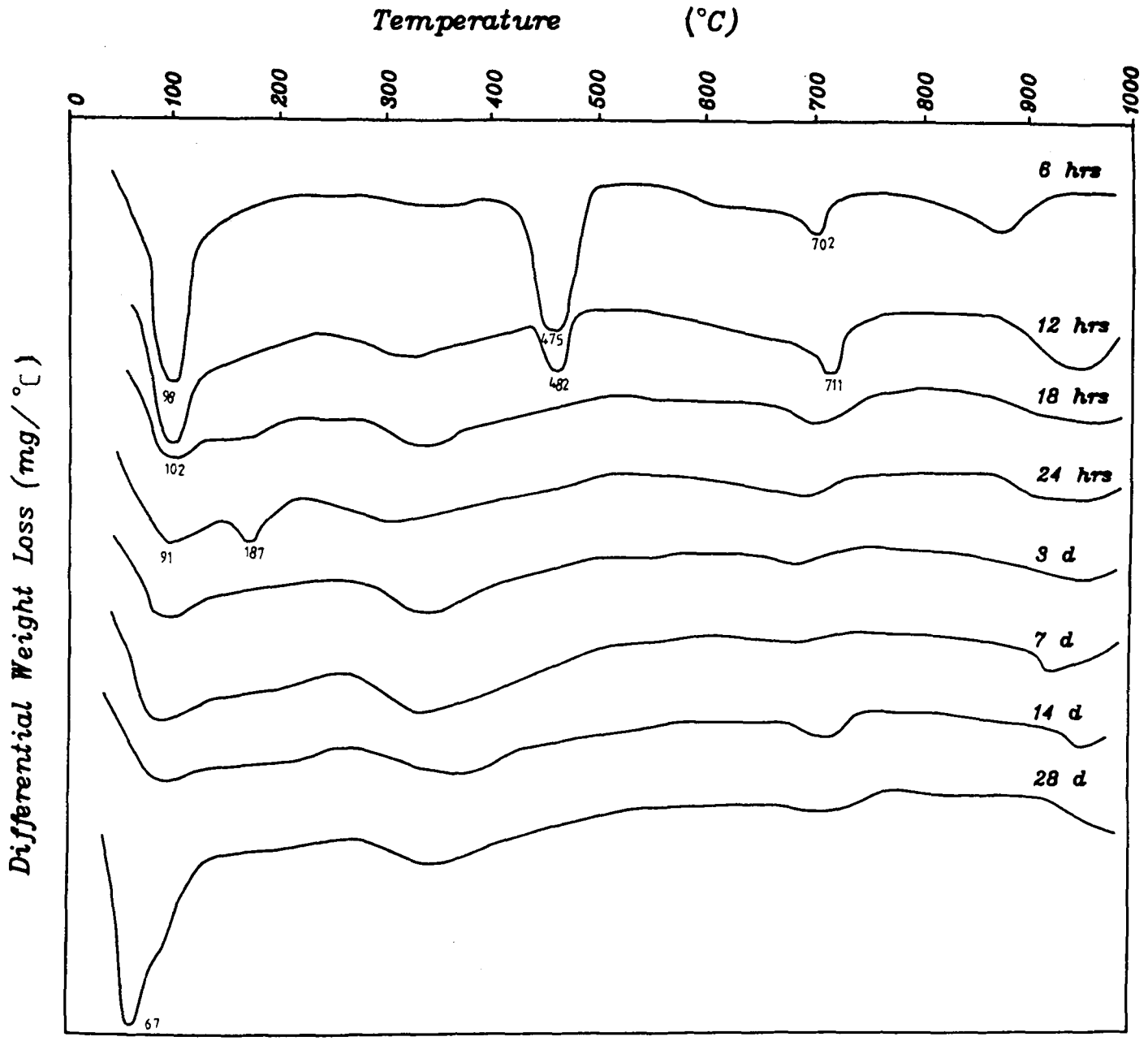


Figure 6.14 DTG thermograms for samples of pfa + 20wt.% lime cured at 95°C and 100% r.h. at curing times from 6 hours to 28 hours.

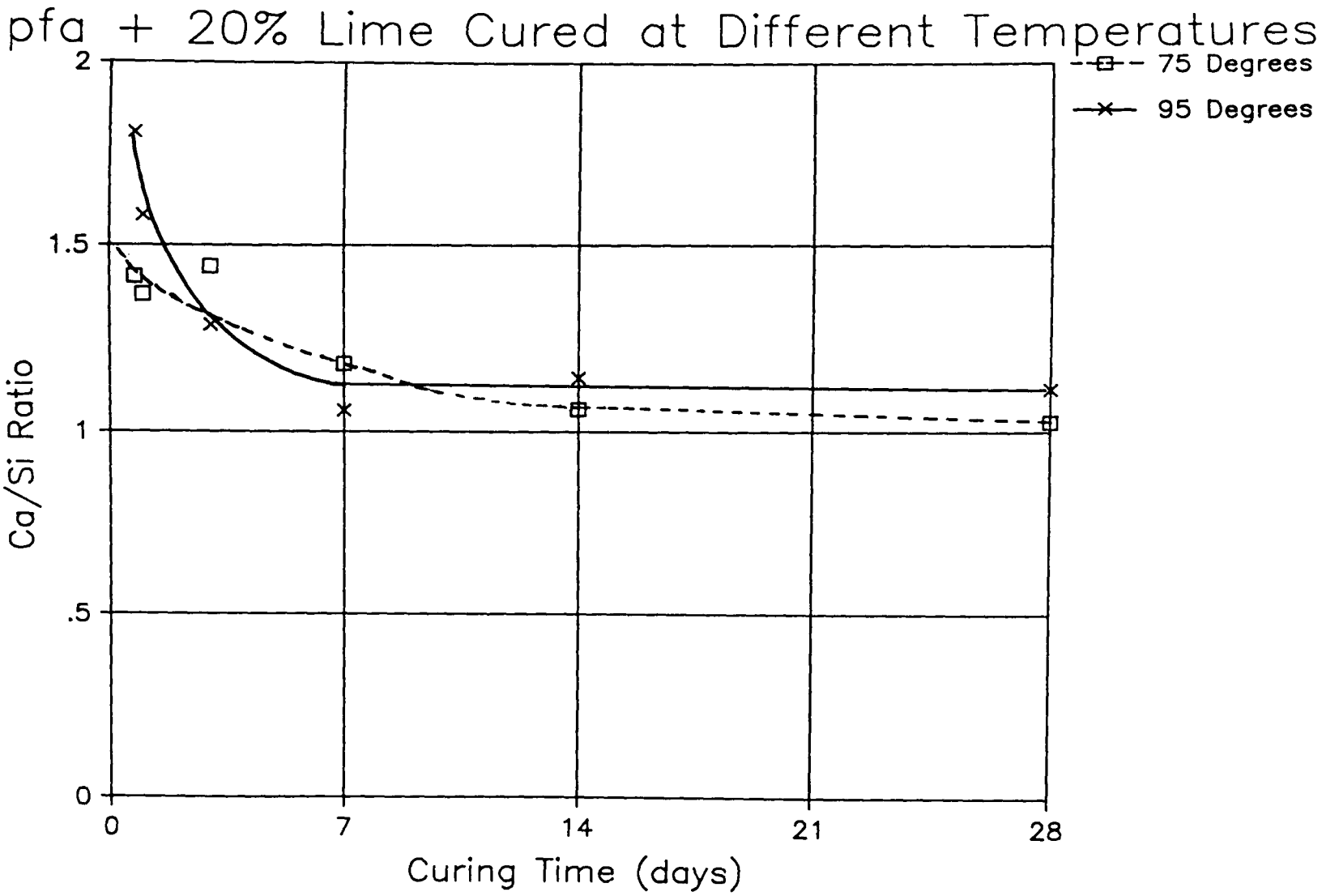


Figure 6.15 Ca/Si ratio versus curing time (up to 28 days) for fibrous gel from pfa + 20wt.% lime cylinders cured at 75°C and 95°C.

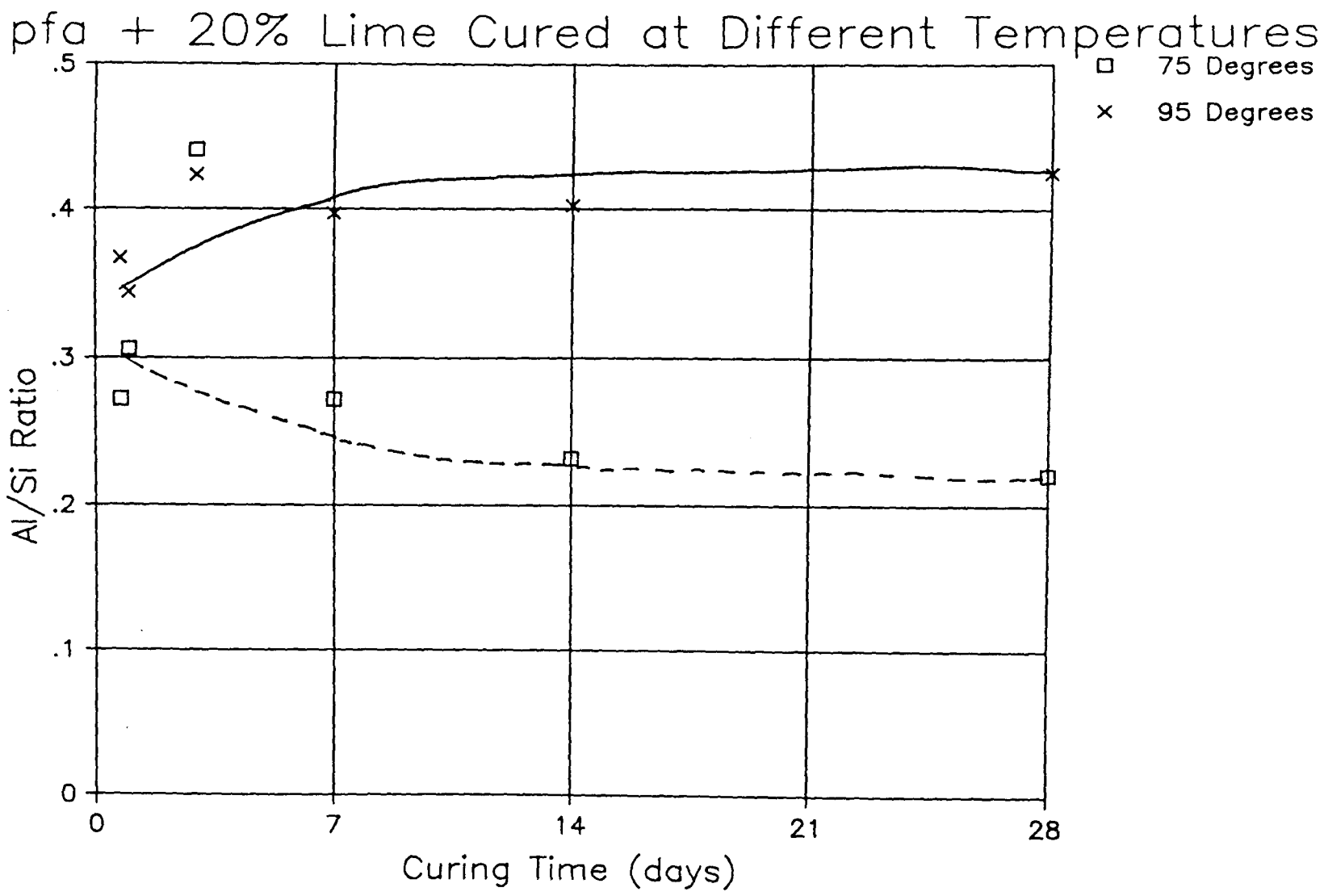
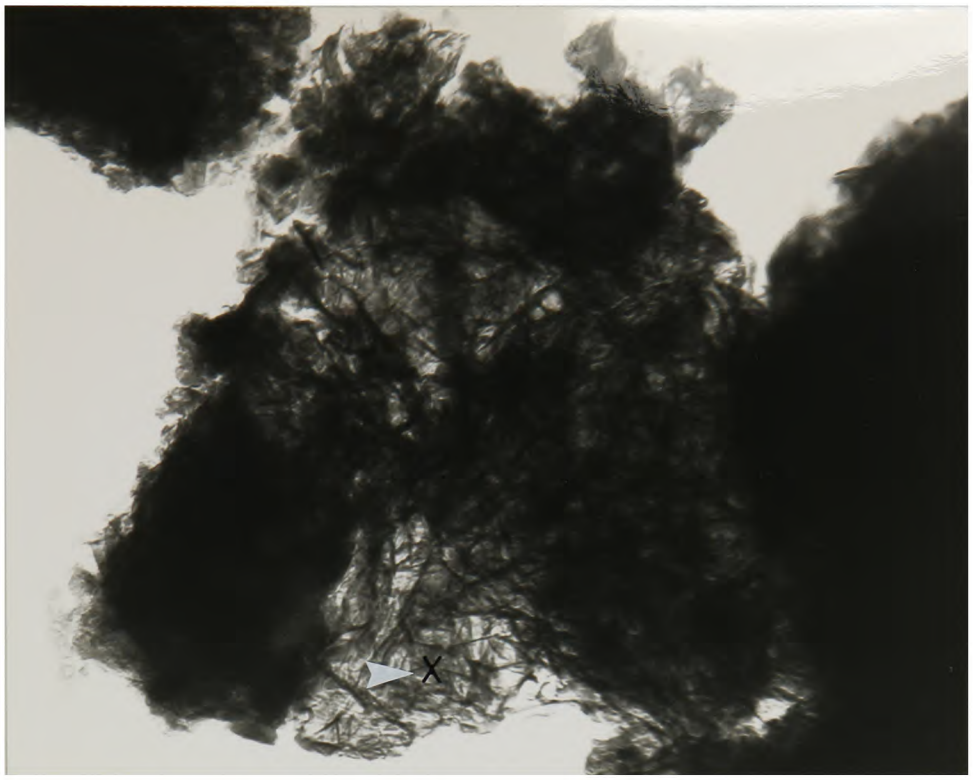


Figure 6.16 Al/Si ratio versus curing time (up to 28 days) for fibrous gel (pfa + 20wt.% lime cylinders cured at 75°C and 95°C).

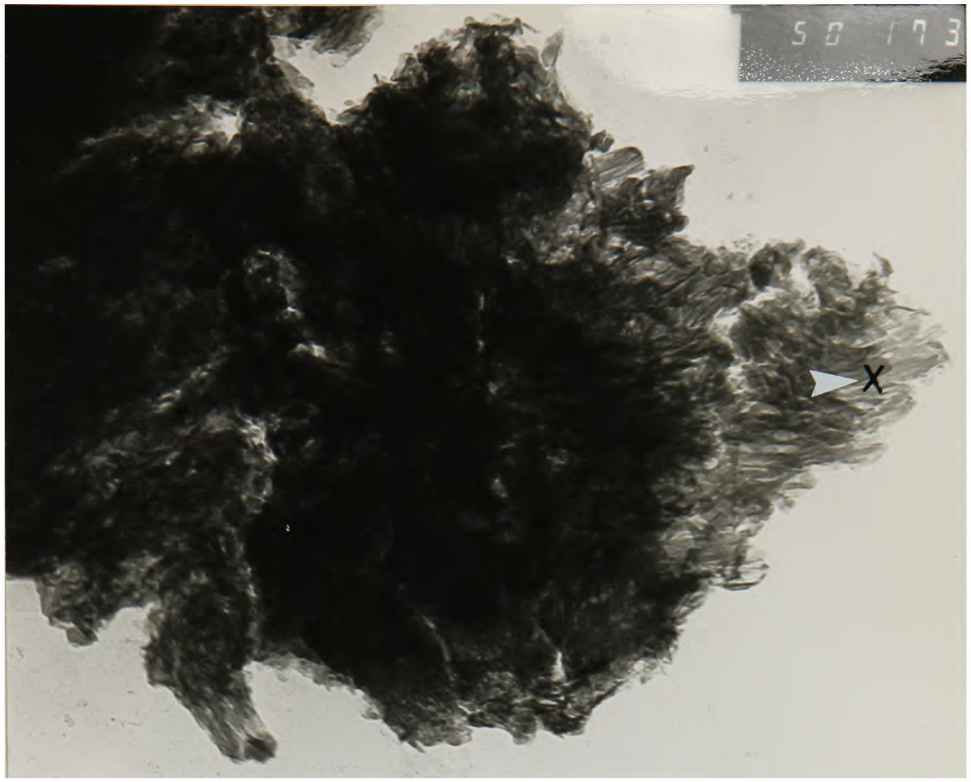


98 nm

EDAX Results

E	%	Ratio to Si
Na	1.36	0.07
Al	5.35	0.29
Si	18.34	-
S	2.49	0.13
K	2.61	0.14
Ca	67.71	3.69
Fe	2.15	0.12

Plate 6.1 TEM micrograph of fibrous gel (pfa + 20wt.% lime specimen cured at 75°C and 100% r.h. for 18 hours).

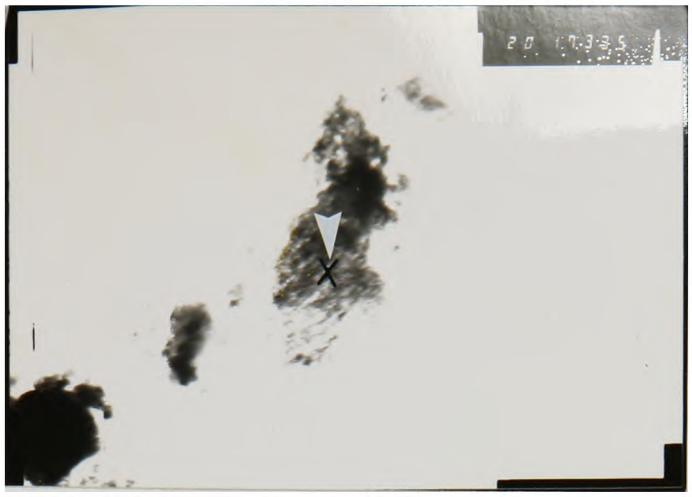


101 nm

EDAX Results

E	%	Ratio to Si
Na	6.88	0.33
Al	7.08	0.34
Si	21.03	-
S	0.92	0.04
K	3.14	0.15
Ca	58.41	2.78
Fe	2.55	0.12

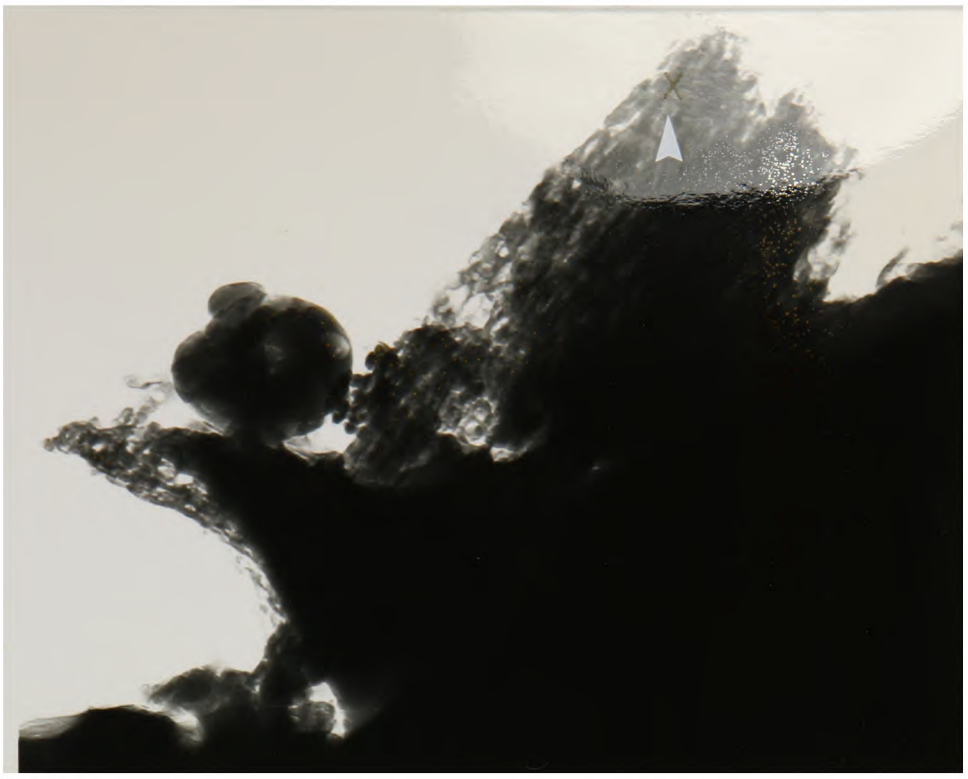
Plate 6.2 TEM micrograph of dense fibrous gel (pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 18 hours).



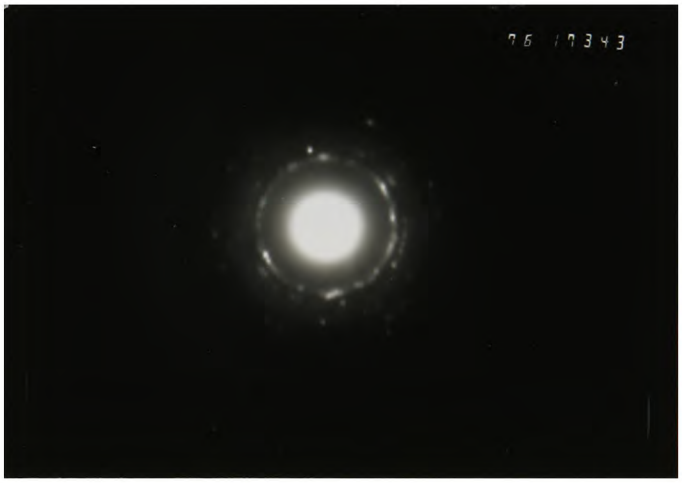
EDAX Results

E	%	Ratio to Si
Na	2.13	0.13
Al	5.77	0.35
Si	16.57	-
S	0.57	0.03
K	1.38	0.08
Ca	72.32	4.37
Fe	1.28	0.08

Plate 6.3 TEM micrograph of fibrous gel and its associated diffraction pattern - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 18 hours.



123 nm



131nm

EDAX Results

E	%	Ratio to Si
Na	3.80	0.44
Al	2.86	0.33
Si	8.54	-
S	0.78	0.09
K	2.93	0.34
Ca	78.23	9.16
Fe	2.97	0.15

Plate 6.4 TEM micrograph of reaction product - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 12 hours. The associated diffraction pattern shows diffraction spots at $\underline{d}$ = 0.280, 0.249, 0.269, 0.202, 0.187, 0.162 and 0.146 (nm).

123nm

EDAX Results

E	%	Ratio to Si
Na	7.12	0.22
Al	14.5	0.45
Si	31.9	-
S	1.66	0.05
K	2.63	0.08
Ca	37.04	1.13
Fe	6.0	0.19

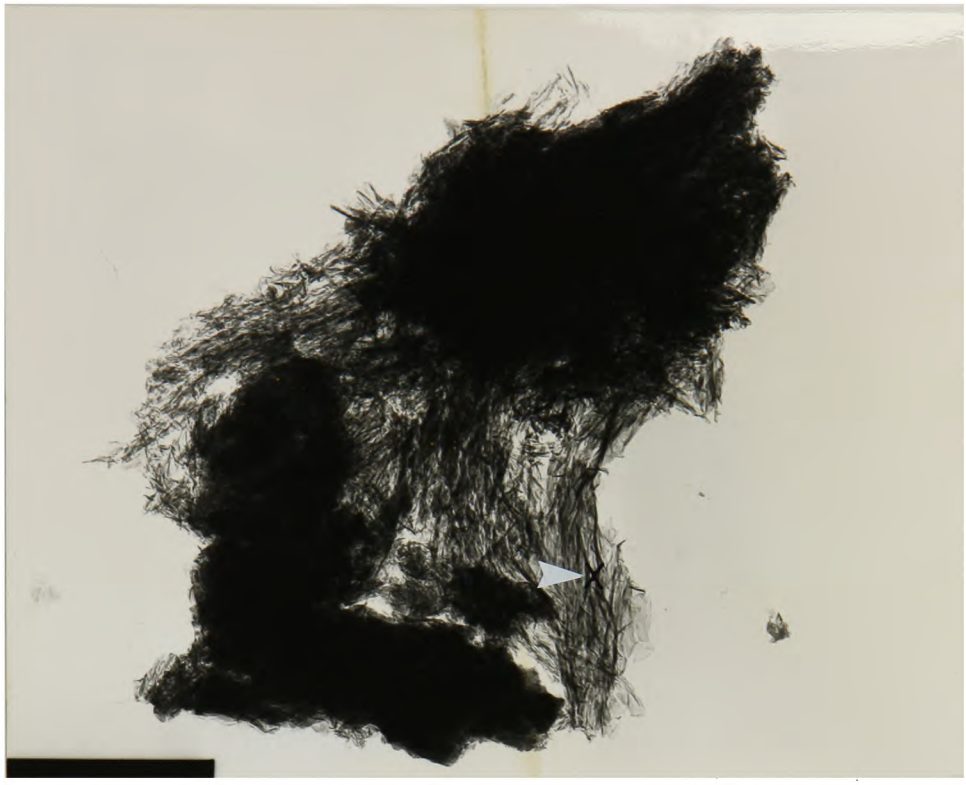
Plate 6.5 TEM micrograph of reaction product - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 12 hours.

183 nm

EDAX Results

E	%	Ratio to Si
Na	-	-
Al	16.5	0.43
Si	38.80	-
S	0.76	0.02
K	0.85	0.02
Ca	39.71	1.02
Fe	3.35	0.09

Plate 6.6 TEM micrograph of open fibrous gel - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 18 hours.

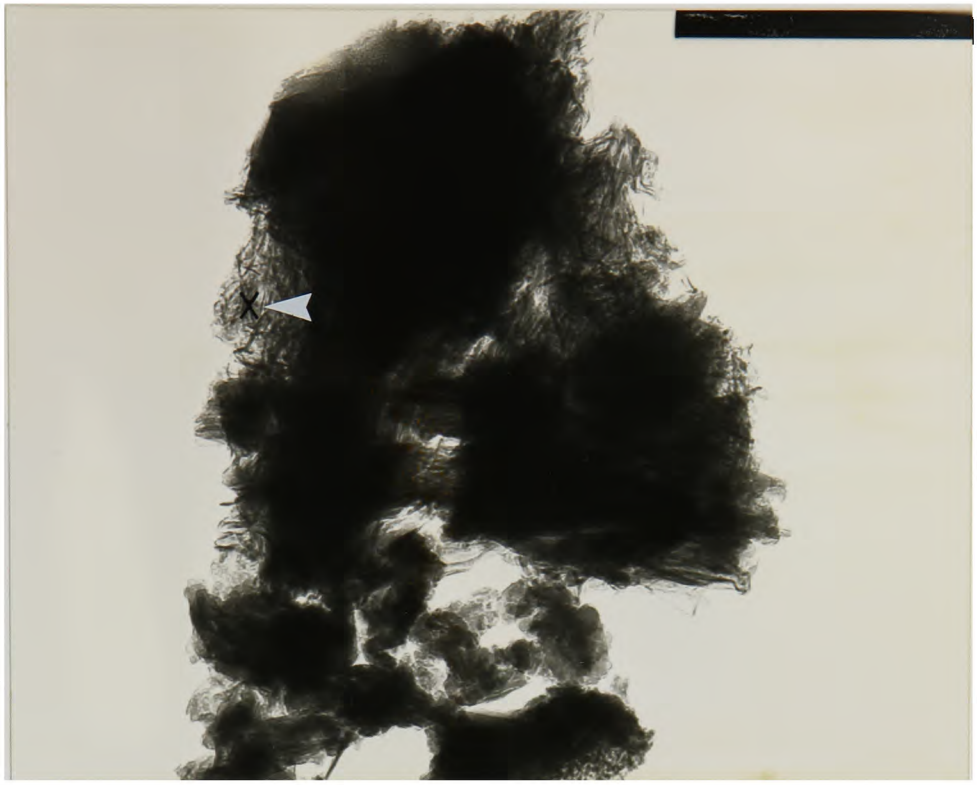


254nm

EDAX Results

E	%	Ratio to Si
Na	0.15	-
Al	13.4	0.36
Si	37.3	-
S	1.02	0.03
K	5.80	0.16
Ca	31.5	0.85
Fe	2.75	0.07

Plate 6.7 TEM micrograph of fibrous gel - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 3 days.

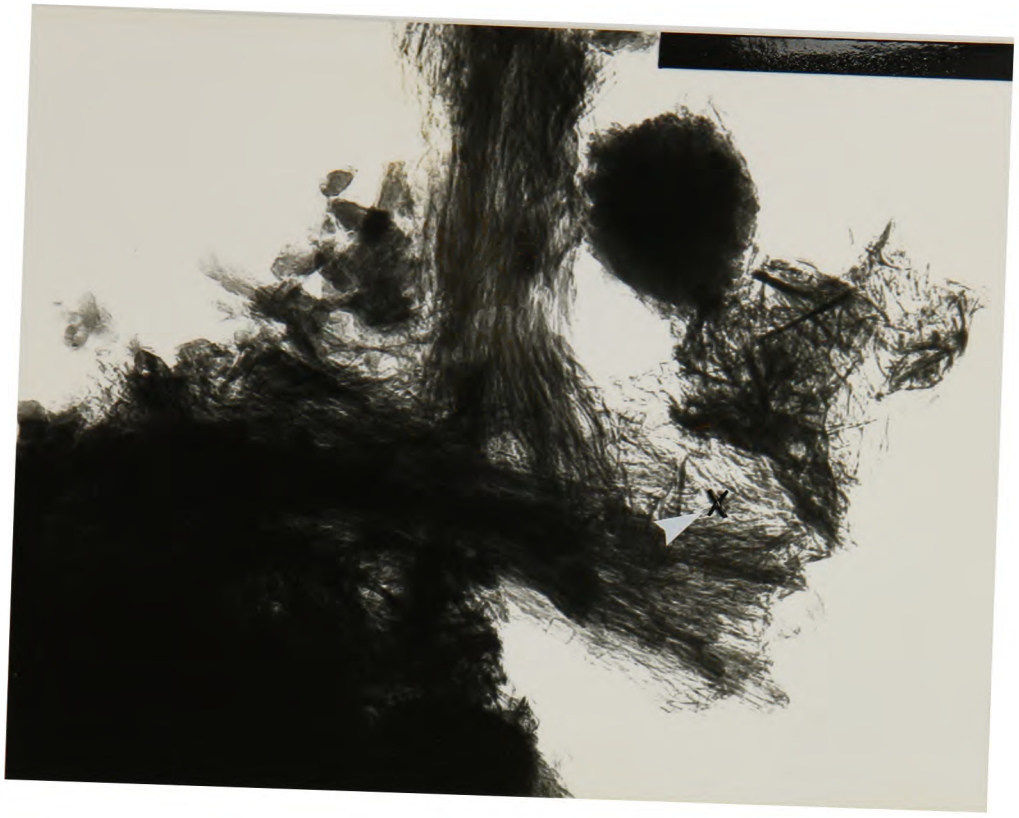


155 nm

EDAX Results

E	%	Ratio to Si
Na	6.59	0.19
Al	14.32	0.24
Si	35.47	-
S	1.14	0.03
K	5.03	0.14
Ca	34.99	0.99
Fe	1.65	0.05

Plate 6.8 TEM micrograph of fibrous gel - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 14 days.



150 nm

EDAX Results

E	%	Ratio to Si
Na	4.88	0.14
Al	13.8	0.4
Si	34.7	-
S	1.63	0.05
K	1.99	0.06
Ca	39.7	1.14
Fe	3.2	0.09

Plate 6.9 TEM micrograph of fibrous gel - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 28 days.

94nm

EDAX Results

E	%	Theoretical ettringite composition (%)
Na	0.75	-
Al	12.83	18.2
Si	1.69	-
S	11.2	27.3
K	1.27	-
Ca	70.62	54.5
Fe	1.65	-

Plate 6.10 TEM micrograph of ettringite crystals - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 12 hours.

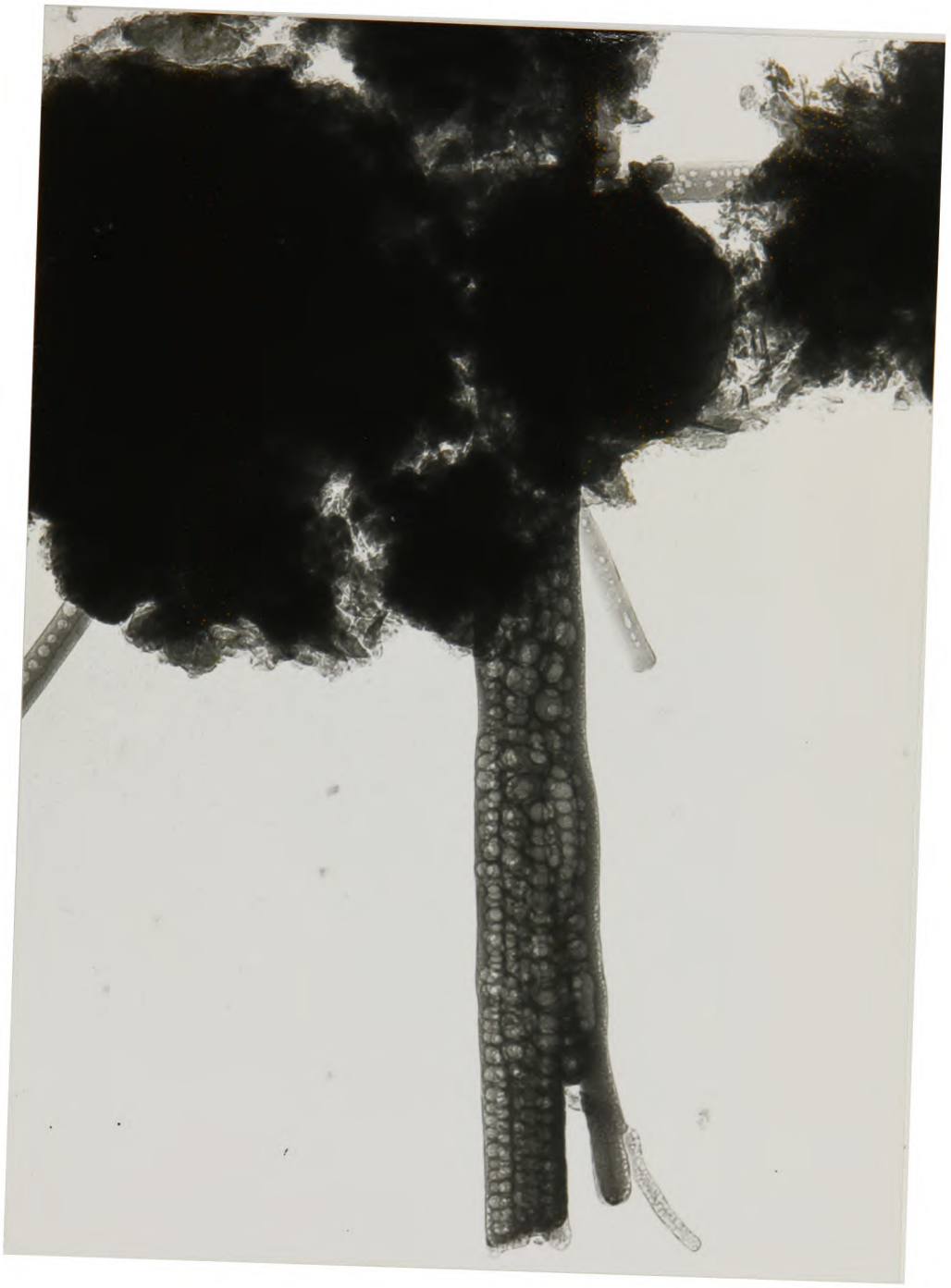


Plate 6.11 TEM micrograph of ettringite crystals - pfa + 20wt.% lime specimen cured at 75°C and 100% r.h. for 3 days.

28nm

EDAX Results

E	%	Theoretical ettringite composition (%)
Na	-	-
Al	16.03	18.2
Si	6.06	-
S	18.92	27.3
K	1.75	-
Ca	54.4	54.5
Fe	2.19	-

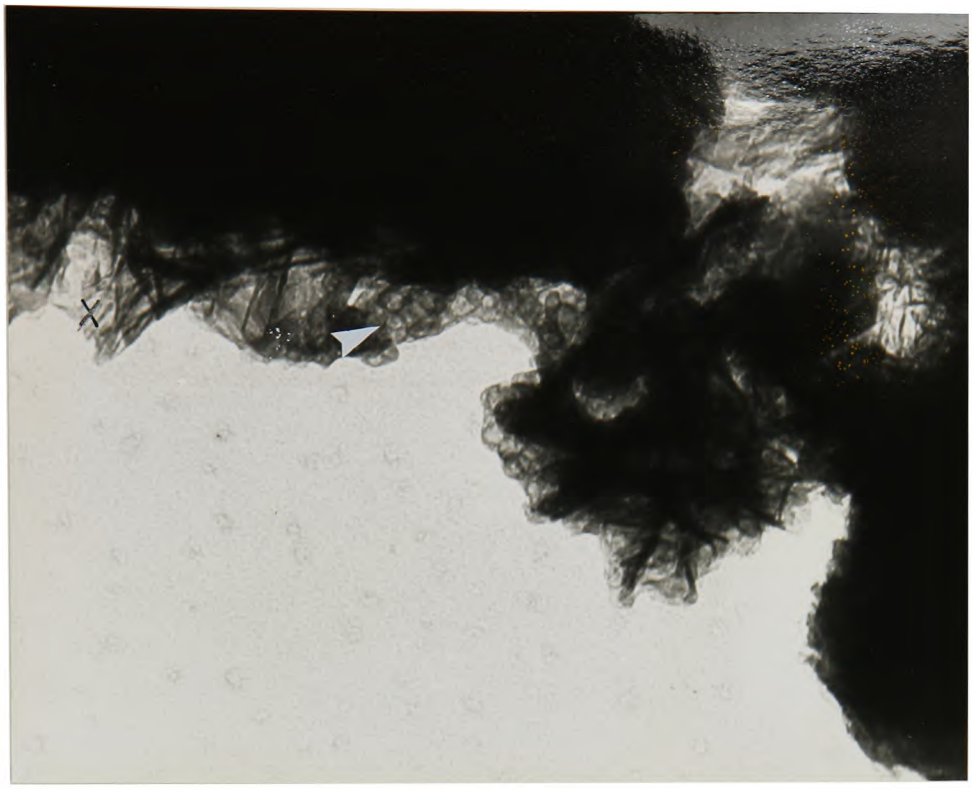
Plate 6.12 TEM micrograph of a specimen made of pfa + 20wt.% lime cured at 75°C and 100% r.h. for 3 days.

106 nm

EDAX Results

E	%	Ratio to Si
Na	-	-
Al	6.34	0.24
Si	26.31	-
S	3.26	0.12
K	2.31	0.09
Ca	89.05	3.38
Fe	2.75	0.10

Plate 6.13 TEM micrograph showing globular material enveloped in fibrous gel - pfa + 20wt.% lime specimen cured at 75°C and 100% r.h. for 24 hours.

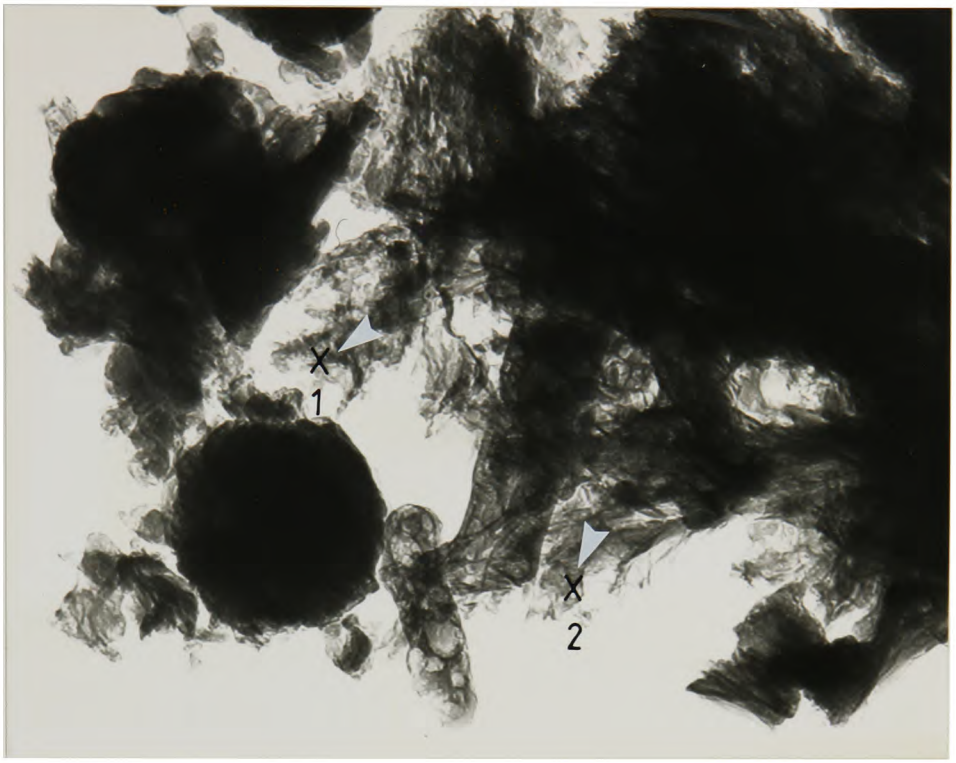


92nm

EDAX Results

E	%	Ratio to Si
Na	-	-
Al	9.56	0.36
Si	29.96	-
S	8.01	0.30
K	6.5	0.24
Ca	43.86	1.64
Fe	5.38	0.20

Plate 6.14 TEM micrograph showing globular product and fibrous gel - pfa + 20wt.% lime specimen cured at 75°C and 100% r.h. for 24 hours.

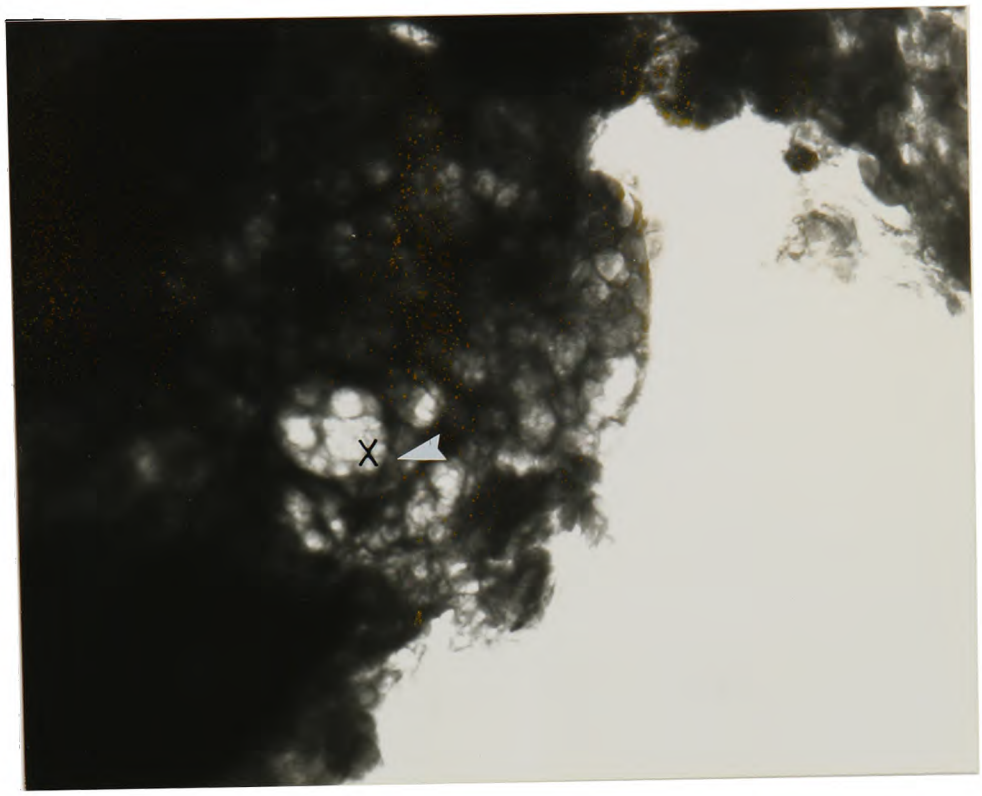


122nm

EDAX Results

E	Analysis 1 %	Analysis 1 Ratio to Si	Analysis 2 %	Analysis 2 Ratio to Si
Na	8.38	0.4	5.12	0.36
Al	14.6	0.69	17.4	1.22
Si	21.1	-	14.24	-
S	7.27	0.34	2.85	0.20
K	5.58	0.17	2.56	0.18
Ca	41.9	1.99	41.47	2.91
Fe	3.1	0.15	5.36	0.37

Plate 6.15 TEM micrograph showing early reaction product - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 12 hours.

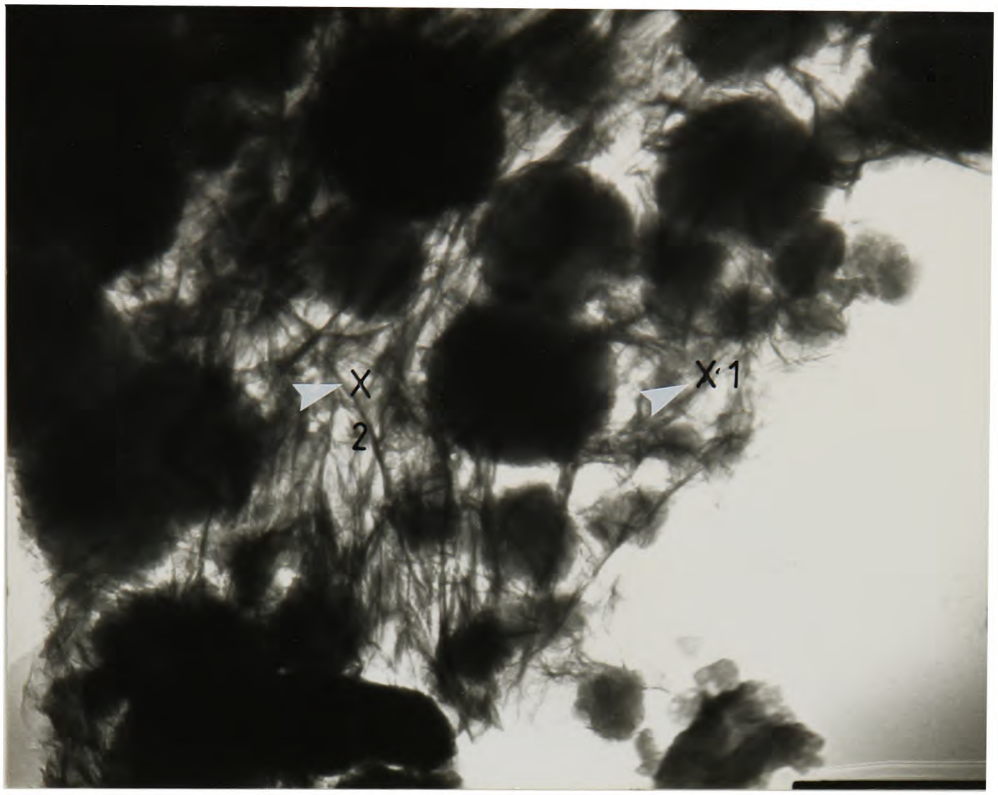


119 nm

EDAX Results

E	%	Theoretical ettringite composition (%)
Na	0.61	-
Al	26.89	18.2
Si	9.82	-
S	8.02	27.3
K	1.26	-
Ca	52.13	54.5
Fe	1.27	-

Plate 6.16 TEM micrograph showing reaction product on a pfa particle - pfa + 20wt.% lime specimen cured at 75°C and 100% r.h. for 3 days.



103 nm

EDAX Results

E	Analysis 1 %	Analysis 1 Ratio to Si	Analysis 2 %	Analysis 2 Ratio to Si
Na	-	-	-	-
Al	10.65	0.29	12.75	0.33
Si	36.66	-	38.75	-
S	0.96	0.03	0.58	0.01
K	4.54	0.12	5.78	0.15
Ca	44.7	1.22	40.0	1.03
Fe	2.42	0.06	2.07	0.05

Plate 6.17 TEM micrograph showing globular product enveloped in fibrous gel - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 7 days.

151 nm

EDAX Results

E	%	Ratio to Si
Na	6.5	0.14
Al	17.6	0.39
Si	45.7	-
S	1.57	0.03
K	7.57	0.17
Ca	18.84	0.41
Fe	2.15	0.05

Plate 6.18 TEM micrograph showing isolated globular particles - pfa + 20wt.% lime specimen cured at 75°C and 100% r.h. for 7 days.

Plate 6.19 SEM micrograph of a specimen of pfa + 20wt.% lime cured at 95°C and 100% r.h. for 3 hours.

Plate 6.20 SEM micrograph of a specimen of pfa + 20wt.% lime cured at 95°C and 100% r.h. for 6 hours.

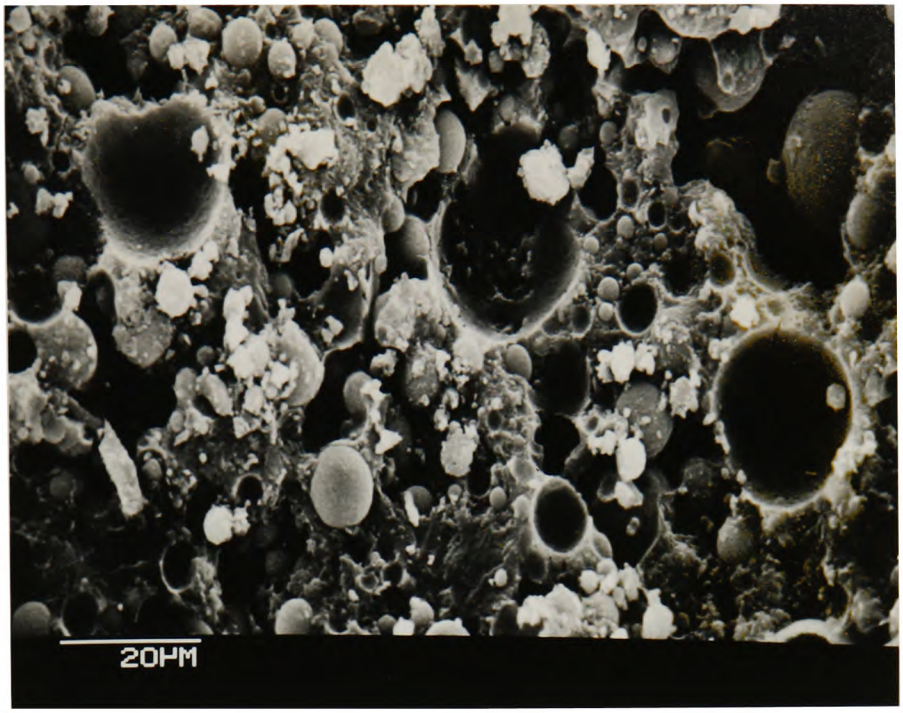


Plate 6.21 SEM micrograph of a specimen of pfa + 20wt.% lime cured at 95°C and 100% r.h. for 12 hours.

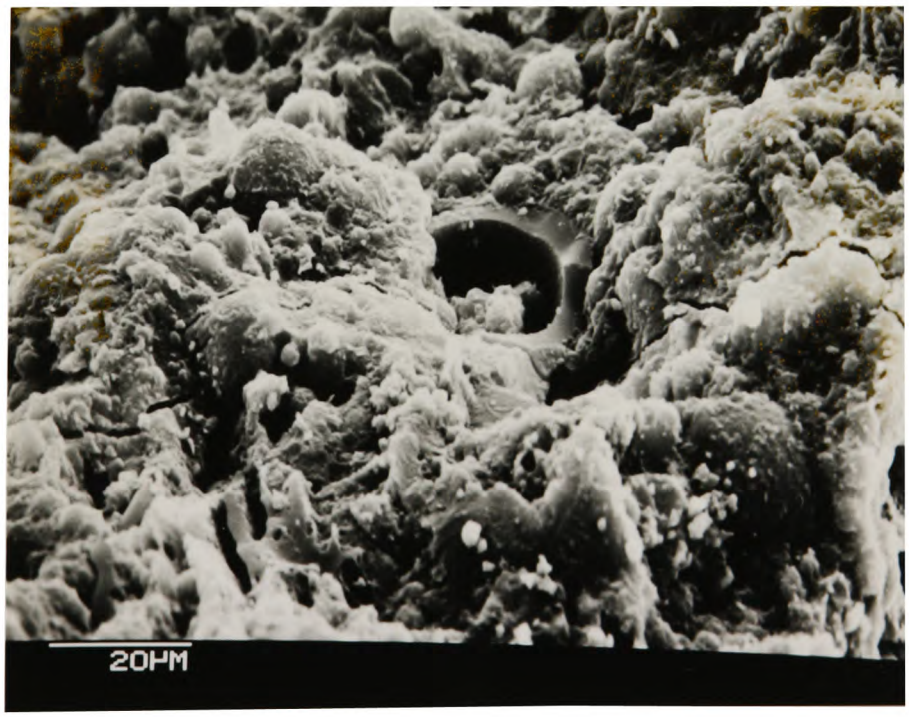


Plate 6.22 SEM micrograph of a specimen of pfa + 20wt.% lime cured at 95°C and 100% r.h. for 18 hours.

Plate 6.23 SEM micrograph of a specimen of pfa + 20wt.% lime cured at 95°C and 100% r.h. for 24 hours.

Plate 6.24 SEM micrograph of a specimen of pfa + 20wt.% lime cured at 95°C and 100% r.h. for 3 days.

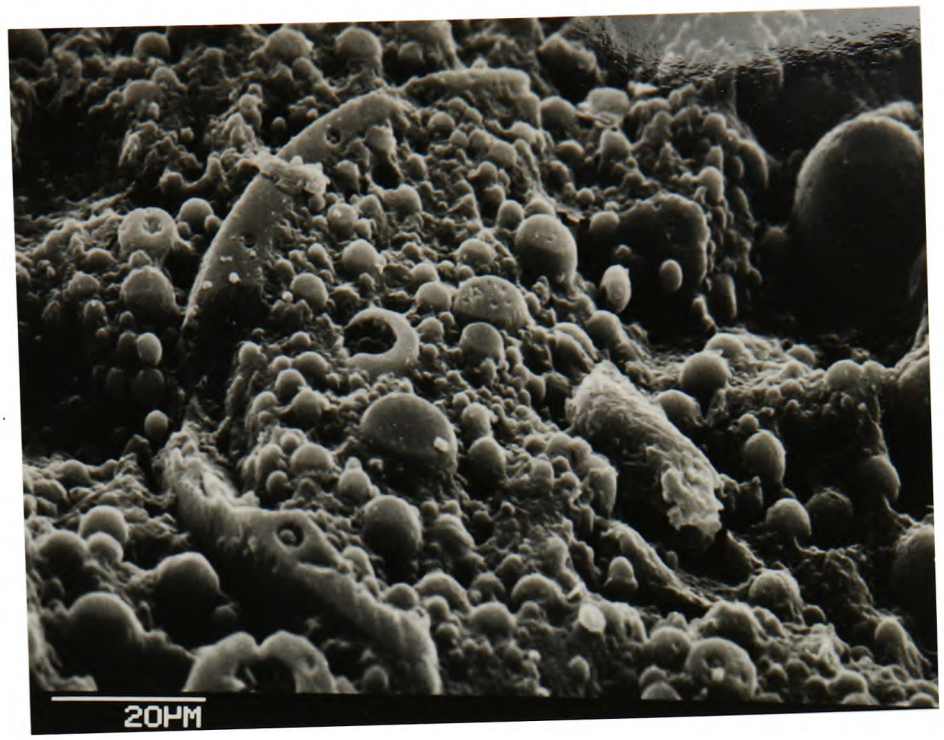


Plate 6.25 SEM micrograph of a specimen of pfa + 20wt.% lime cured at 95°C and 100% r.h. for 7 days.

Plate 6.26 SEM micrograph of a specimen of pfa + 20wt.% lime cured at 95°C and 100% r.h. for 14 days.

Plate 6.27 SEM micrograph of a specimen of pfa + 20wt.% lime cured at 95°C and 100% r.h. for 28 days.

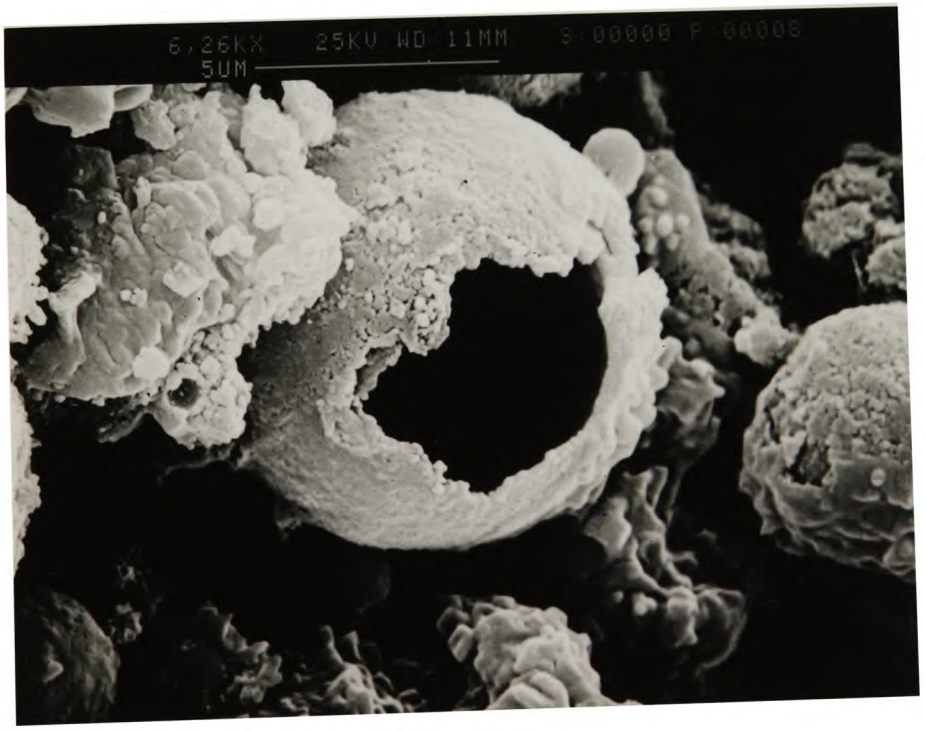


Plate 6.28 SEM micrograph showing a reacted shell - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 3 hours.

Plate 6.29 SEM micrograph showing a part of a reacted shell - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 12 hours.

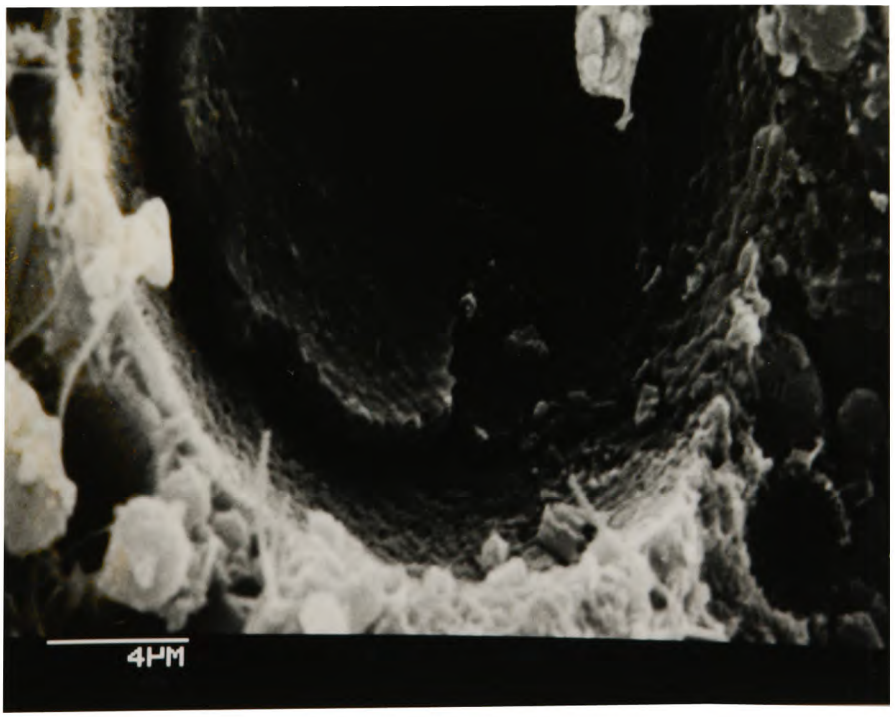


Plate 6.30 SEM micrograph showing a piece of a reacted shell - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 12 hours.

Plate 6.31 Low magnification SEM micrograph showing growth of foil-like gel - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 12 hours.

Plate 6.32 High magnification SEM micrograph showing growth of foil-like gel - pfa + 20wt.% lime specimen cured at 95°C and 100% r.h. for 12 hours.

CHAPTER SEVEN

TABLES, FIGURES AND PLATES

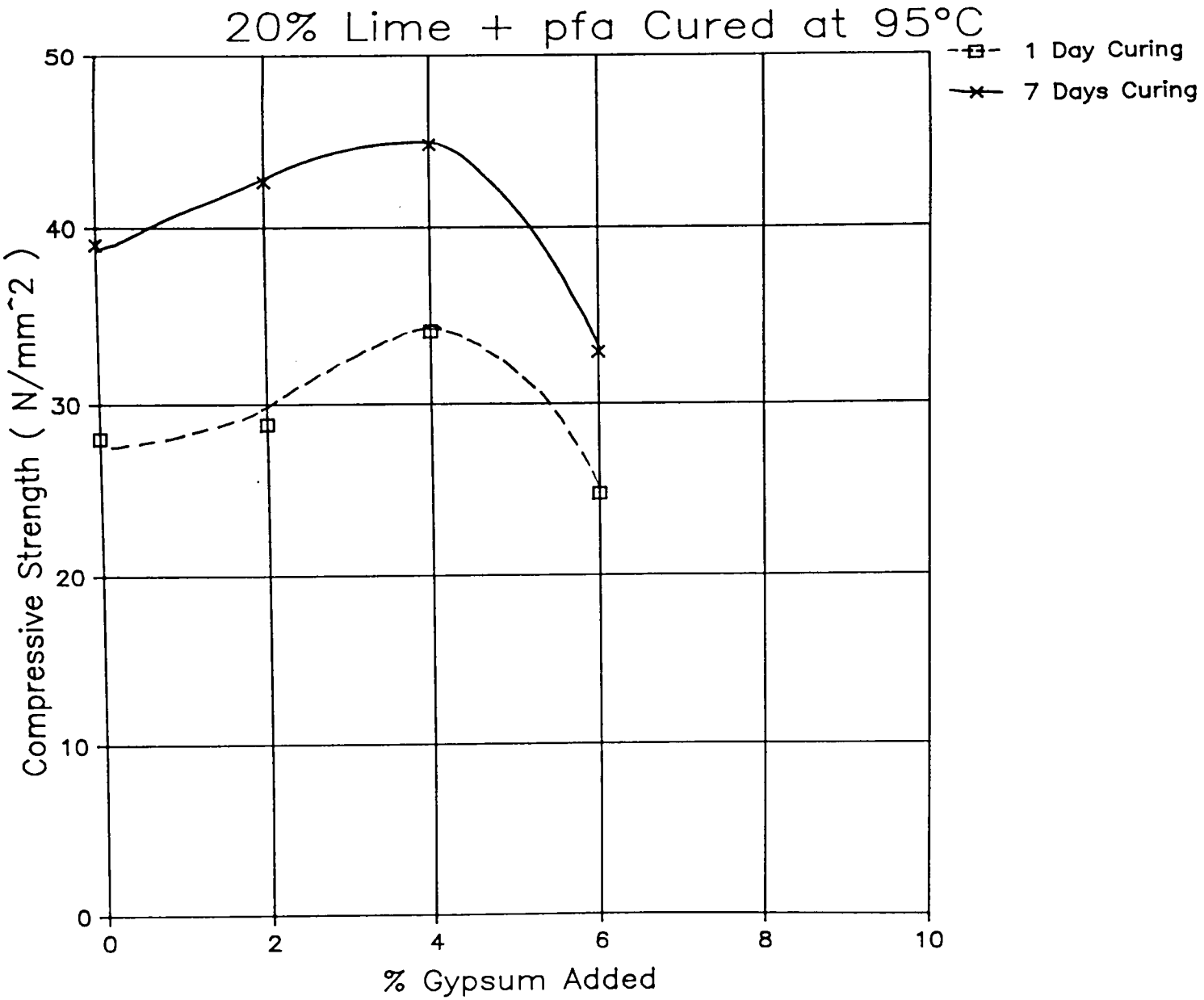


Figure 7.1 Compressive strength versus % gypsum additions - pfa + 20wt.% lime cylinders cured at 95°C and 100% r.h. for 1 and 7 days.

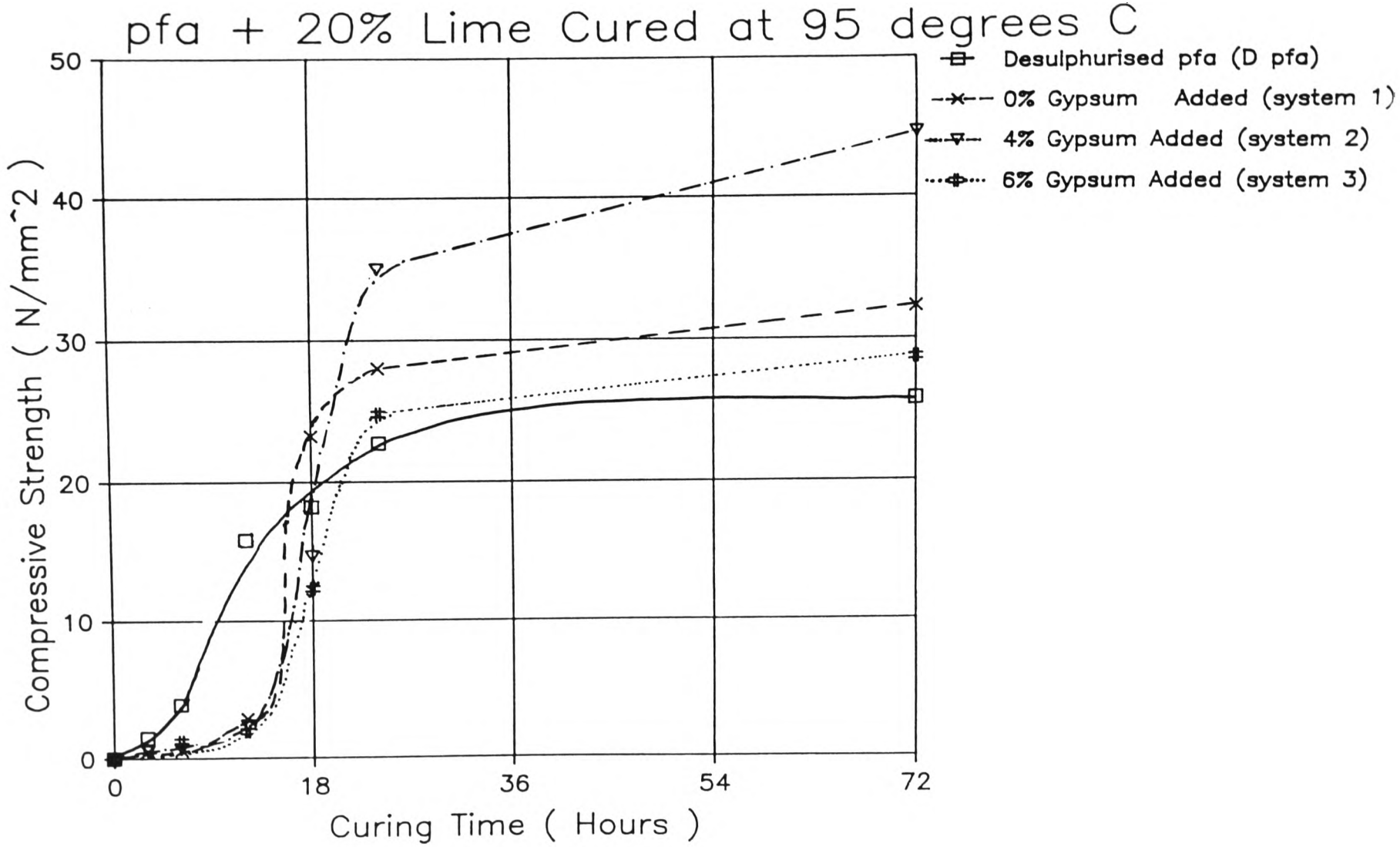


Figure 7.2 Compressive strength versus curing time (up to 3 days) for the D pfa system, system 1, system 2 and system 3.

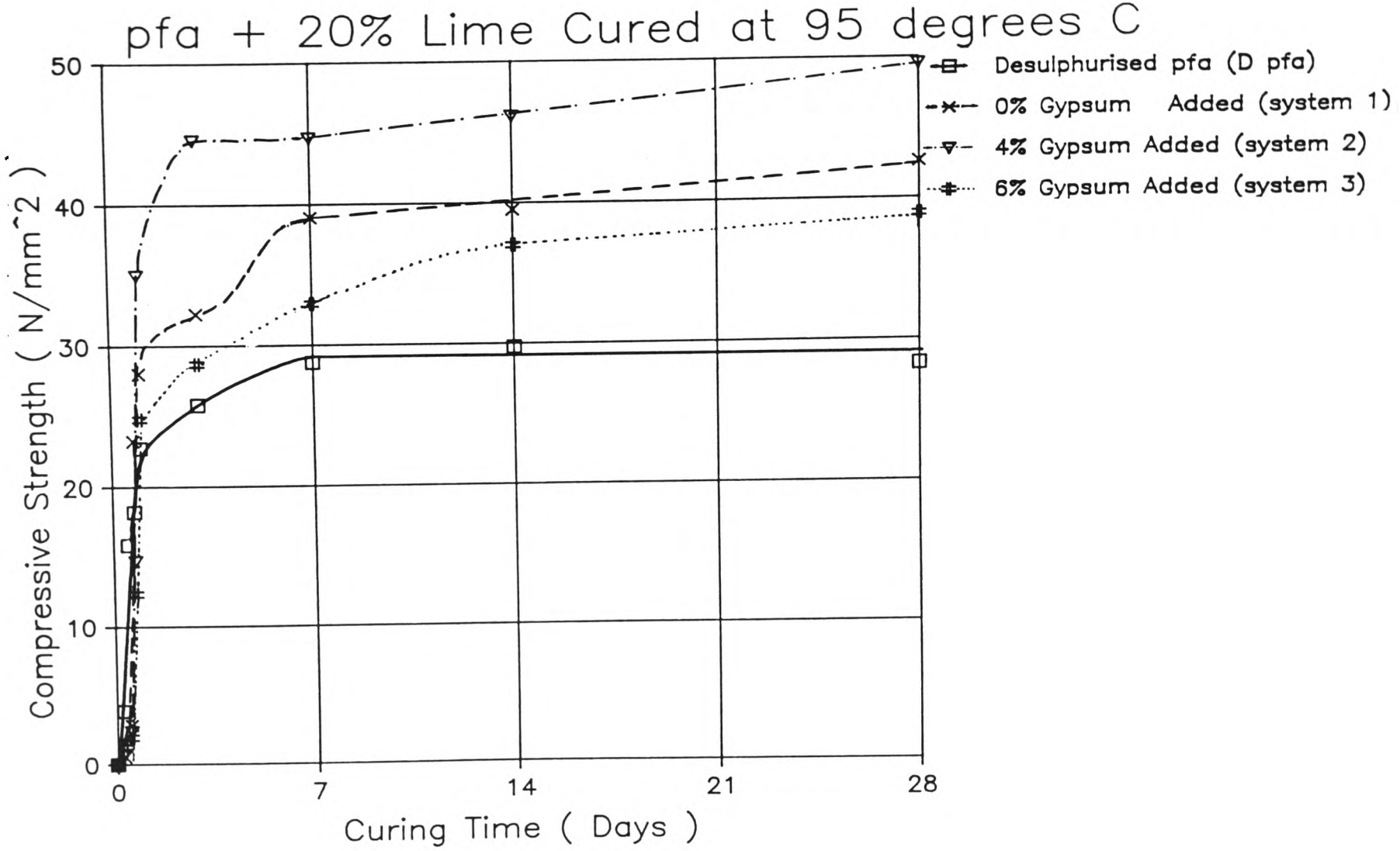


Figure 7.3 Compressive strength versus curing time (up to 28 days) for the D pfa system, system 1, system 2 and system 3.

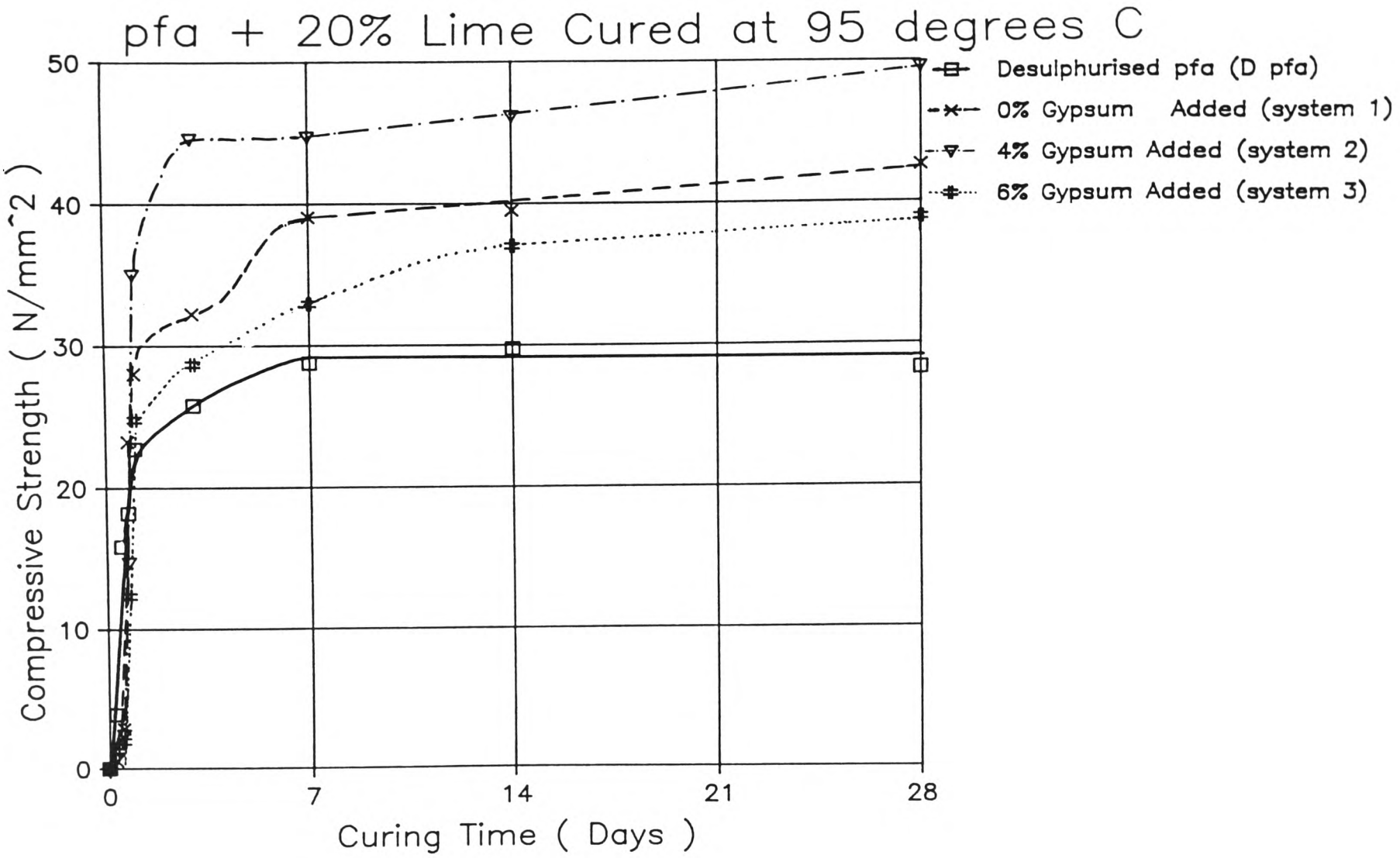


Figure 7.3 Compressive strength versus curing time (up to 28 days) for the D pfa system, system 1, system 2 and system 3.

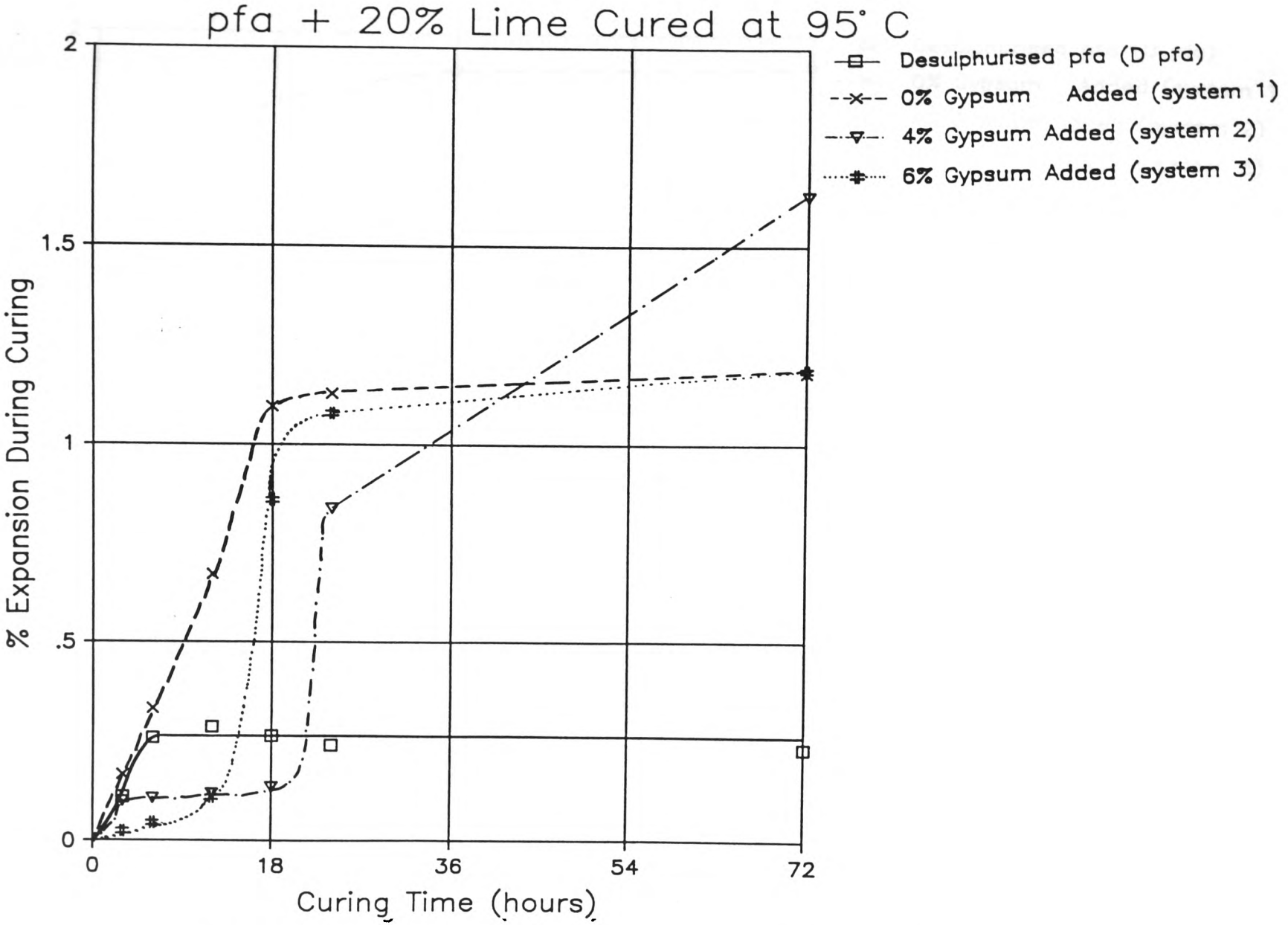


Figure 7.4 Expansion during curing versus curing time (up to 3 days) for the D pfa system, system 1, system 2 and system 3.

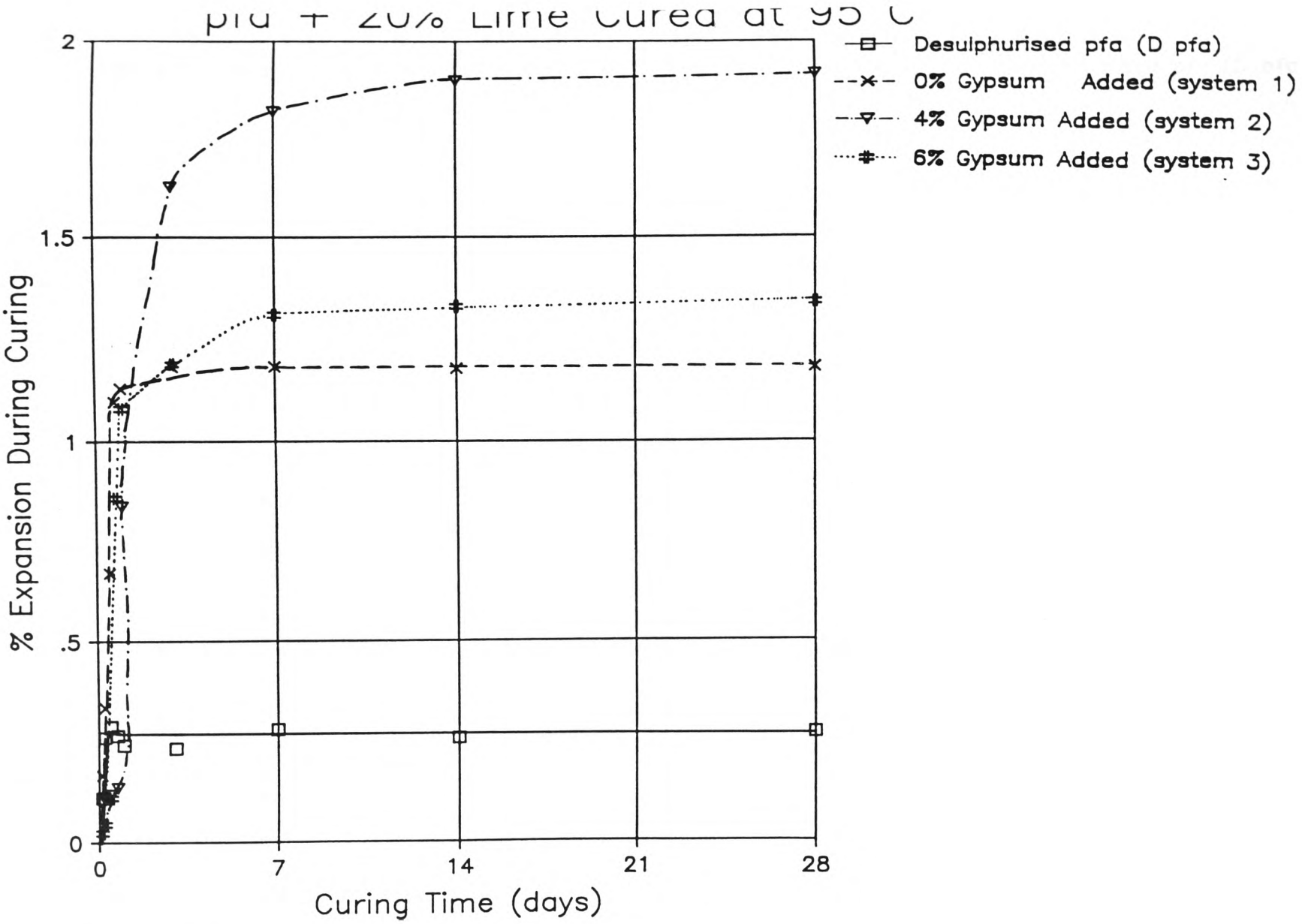


Figure 7.5 Expansion during curing versus curing time (up to 28 days) for the D pfa system, system 1, system 2 and system 3.

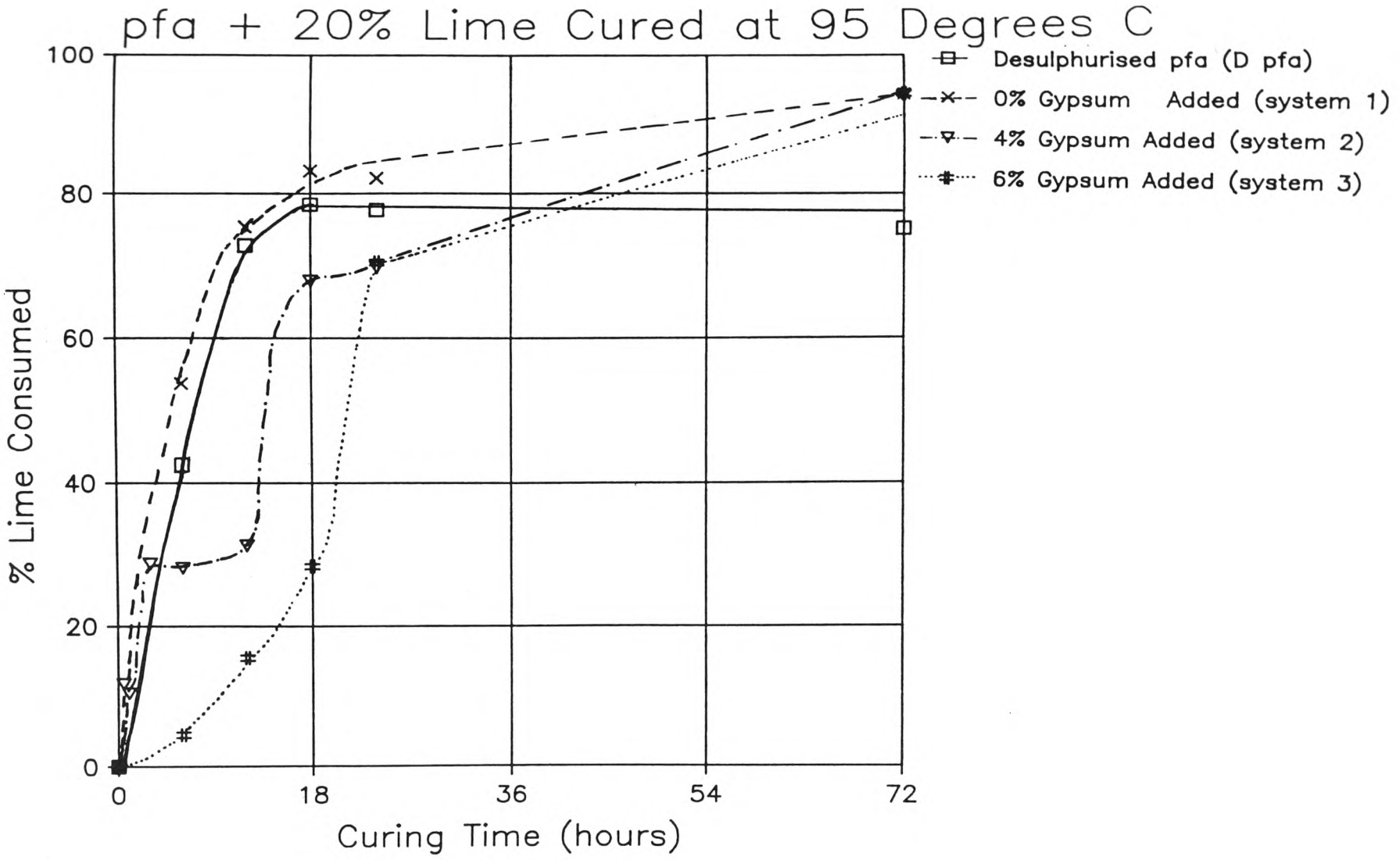


Figure 7.6 Percentage lime consumption (determined using DTG) versus curing time (up to 3 days) for the D pfa system, system 1, system 2 and system 3.

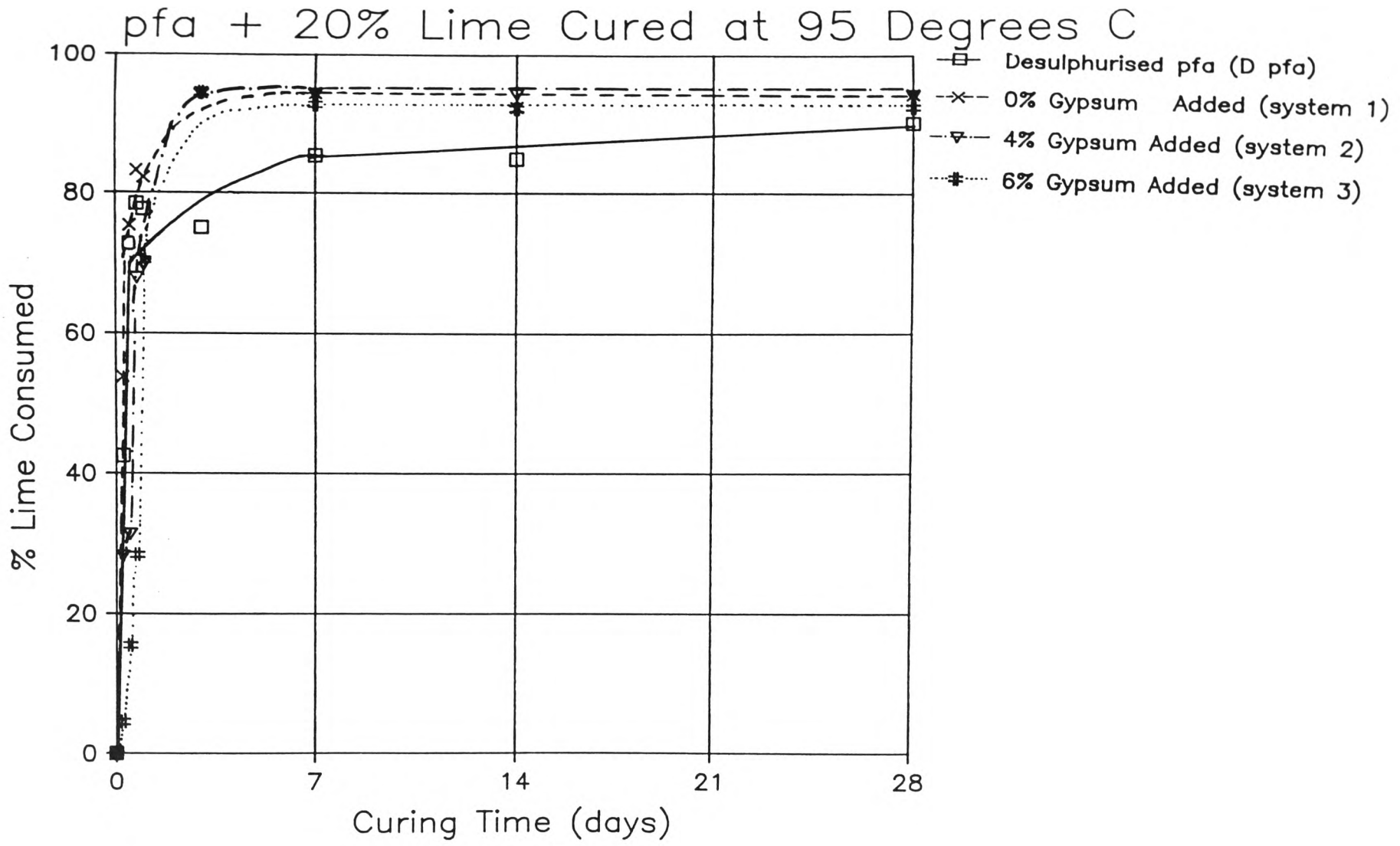


Figure 7.7 Percentage lime consumption (determined using DTG) versus curing time (up to 28 days) for the D pfa system, system 1, system 2 and system 3.

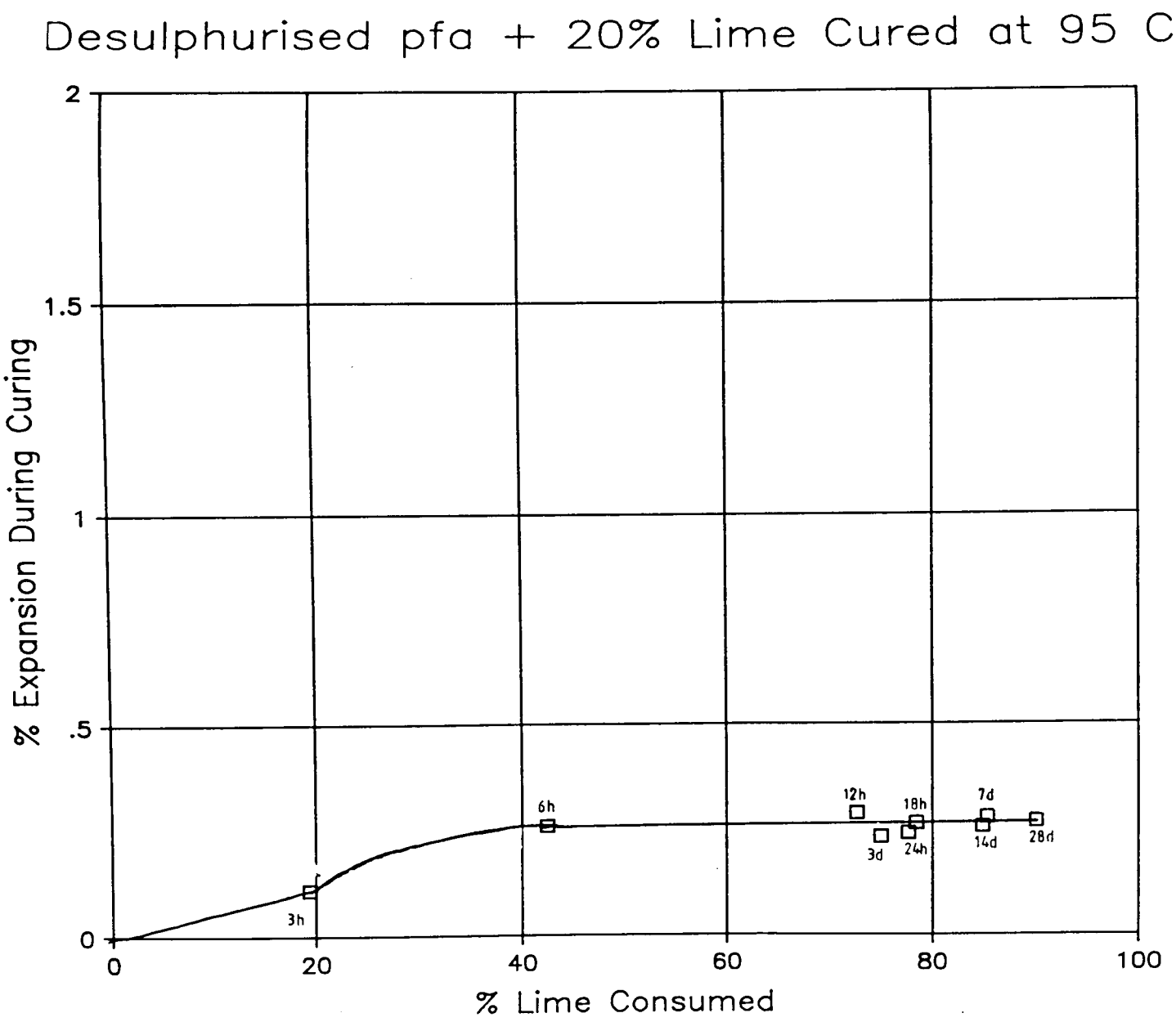


Figure 7.8a Expansion during curing versus % lime consumption for system D pfa cured up to 28 days. (curing time denoted, h = hours, d = days)

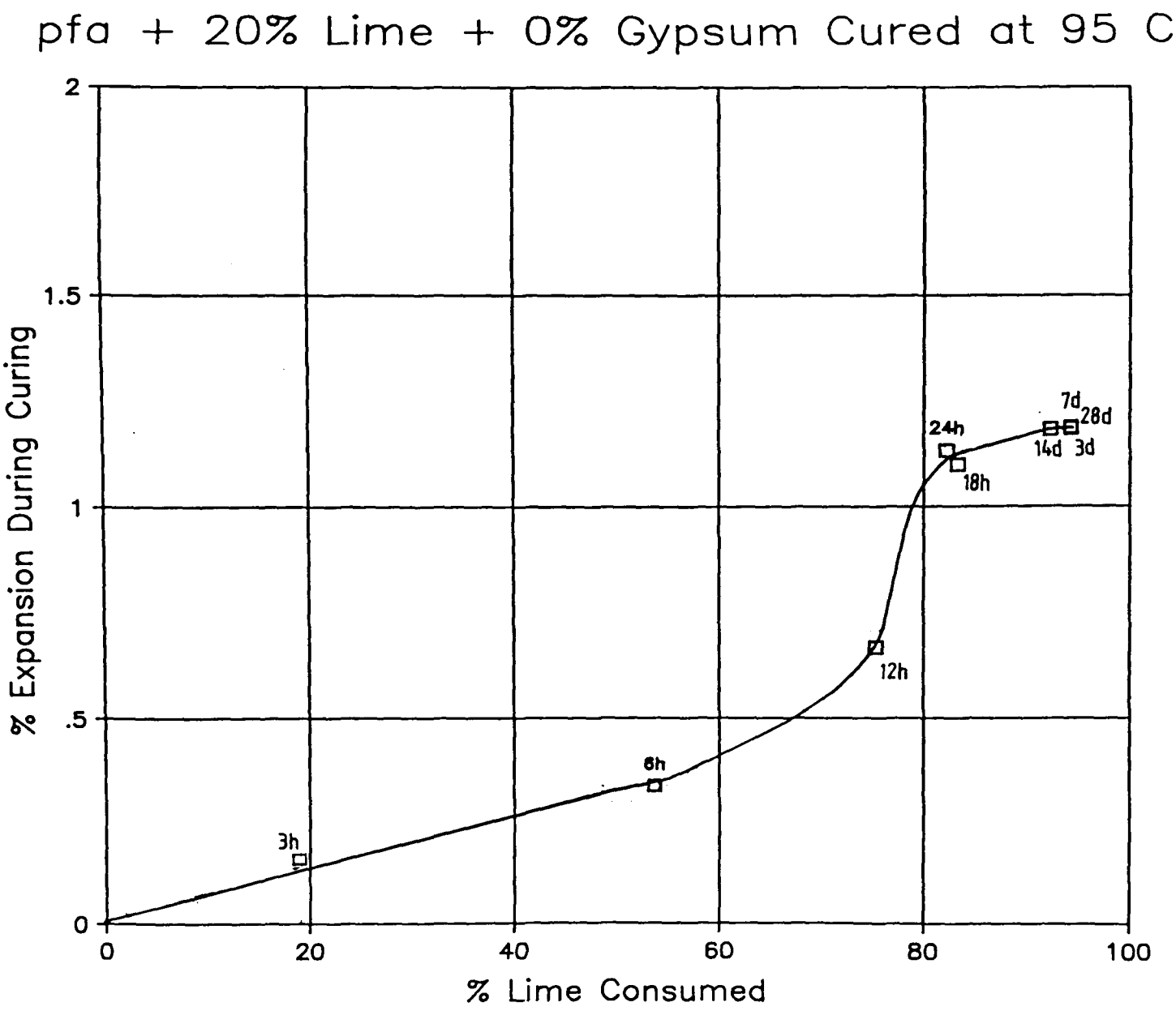


Figure 7.8b Expansion during curing versus % lime consumption for system 1 cured up to 28 days. (curing time denoted, h = hours, d = days)

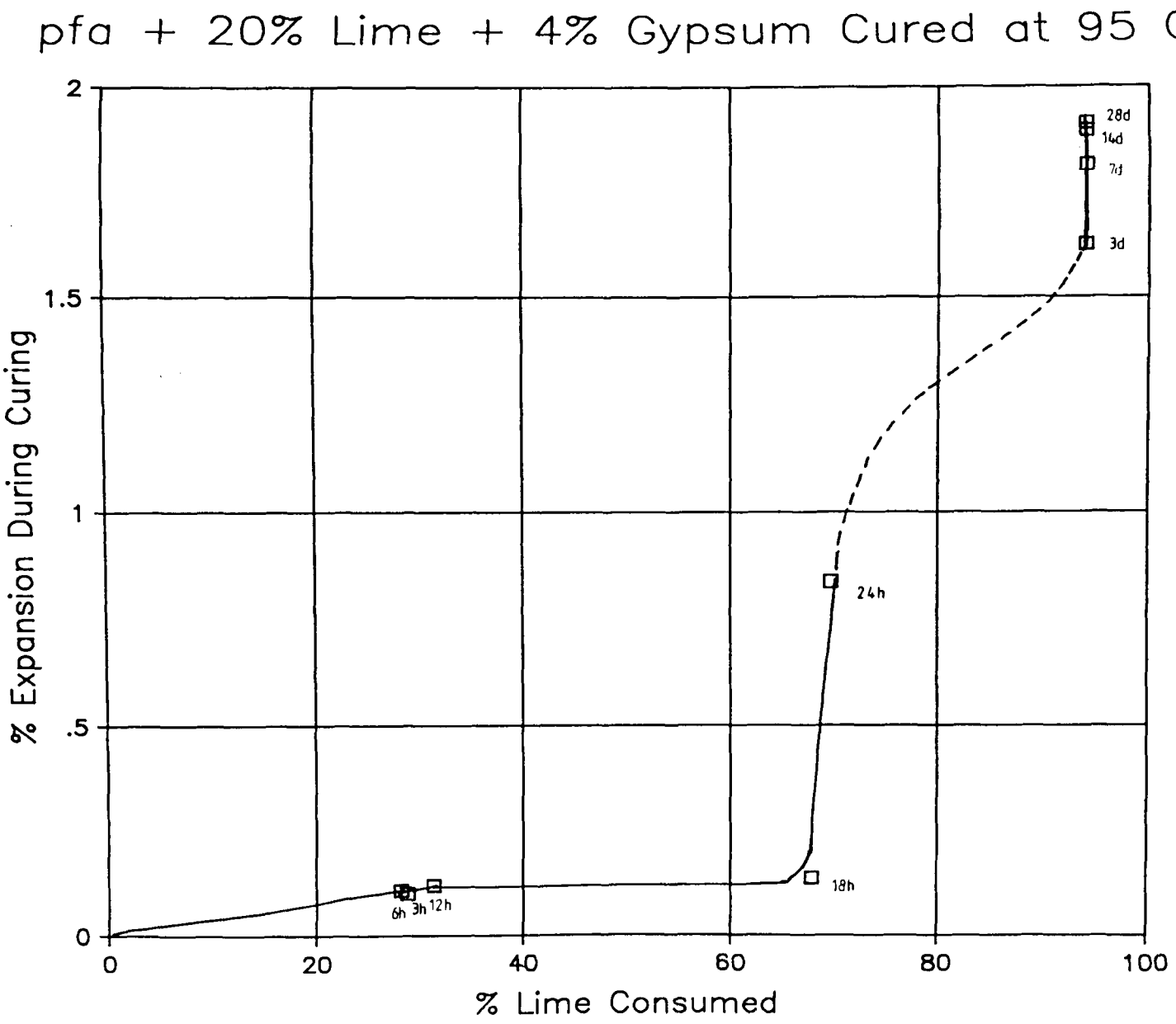


Figure 7.8c Expansion during curing versus % lime consumption for system 2 cured up to 28 days.
(curing time denoted, h = hours, d = days)

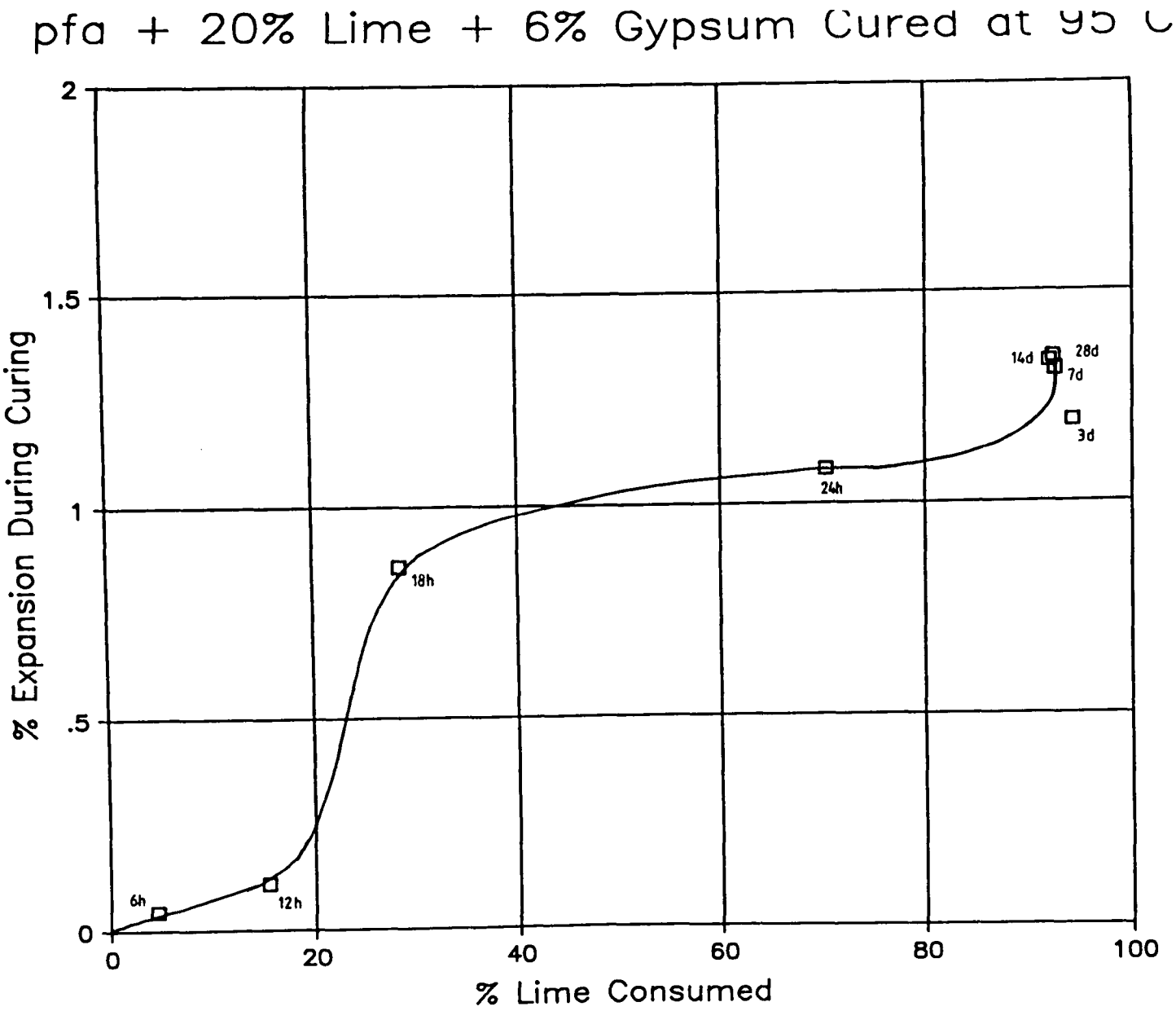


Figure 7.8d Expansion during curing versus % lime consumption for system 3 cured up to 28 days.
(curing time denoted, h = hours, d = days)

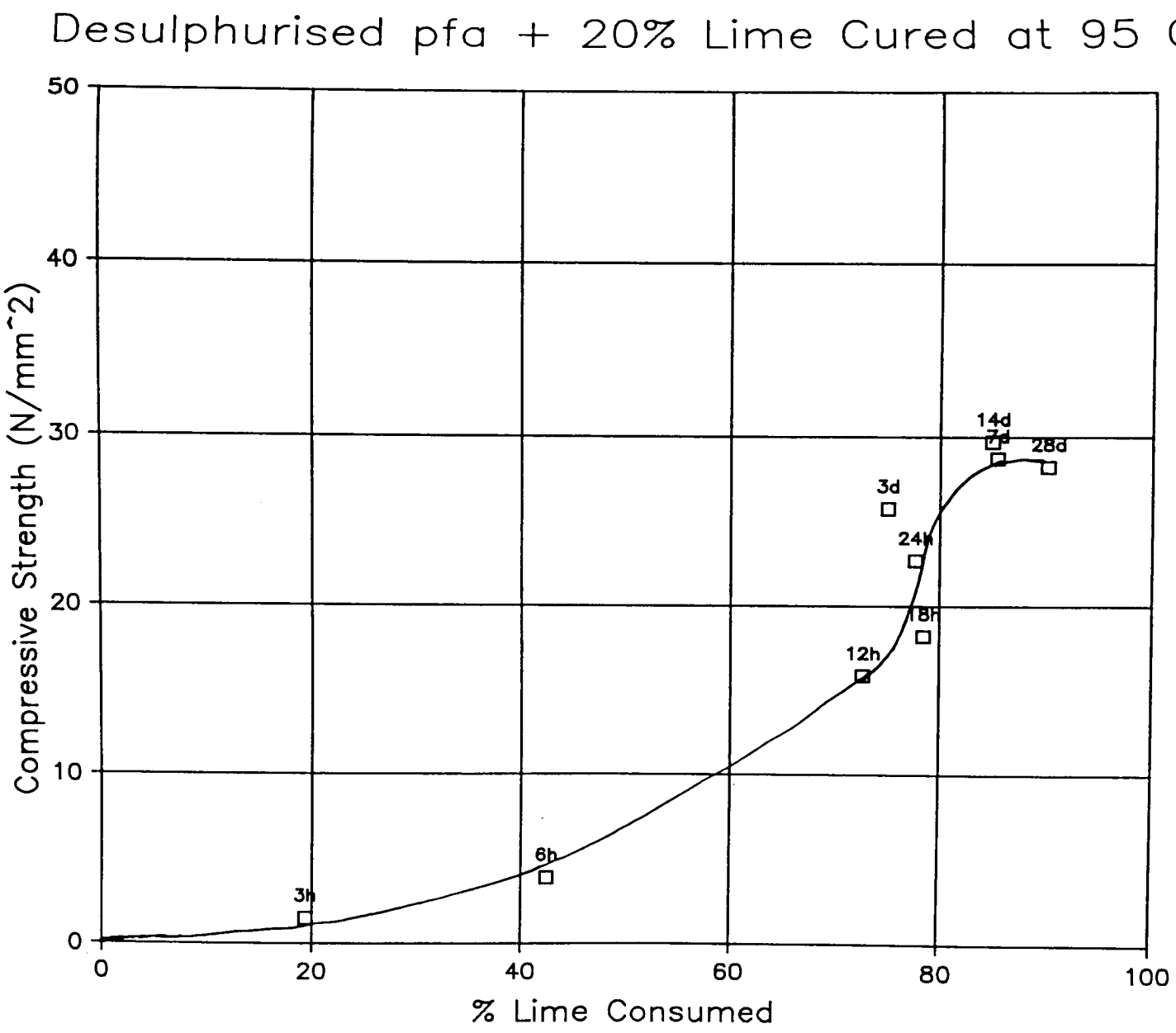


Figure 7.9a Compressive strength versus % lime consumption for system D pfa cured up to 28 days. (curing time denoted, h = hours, d = days)

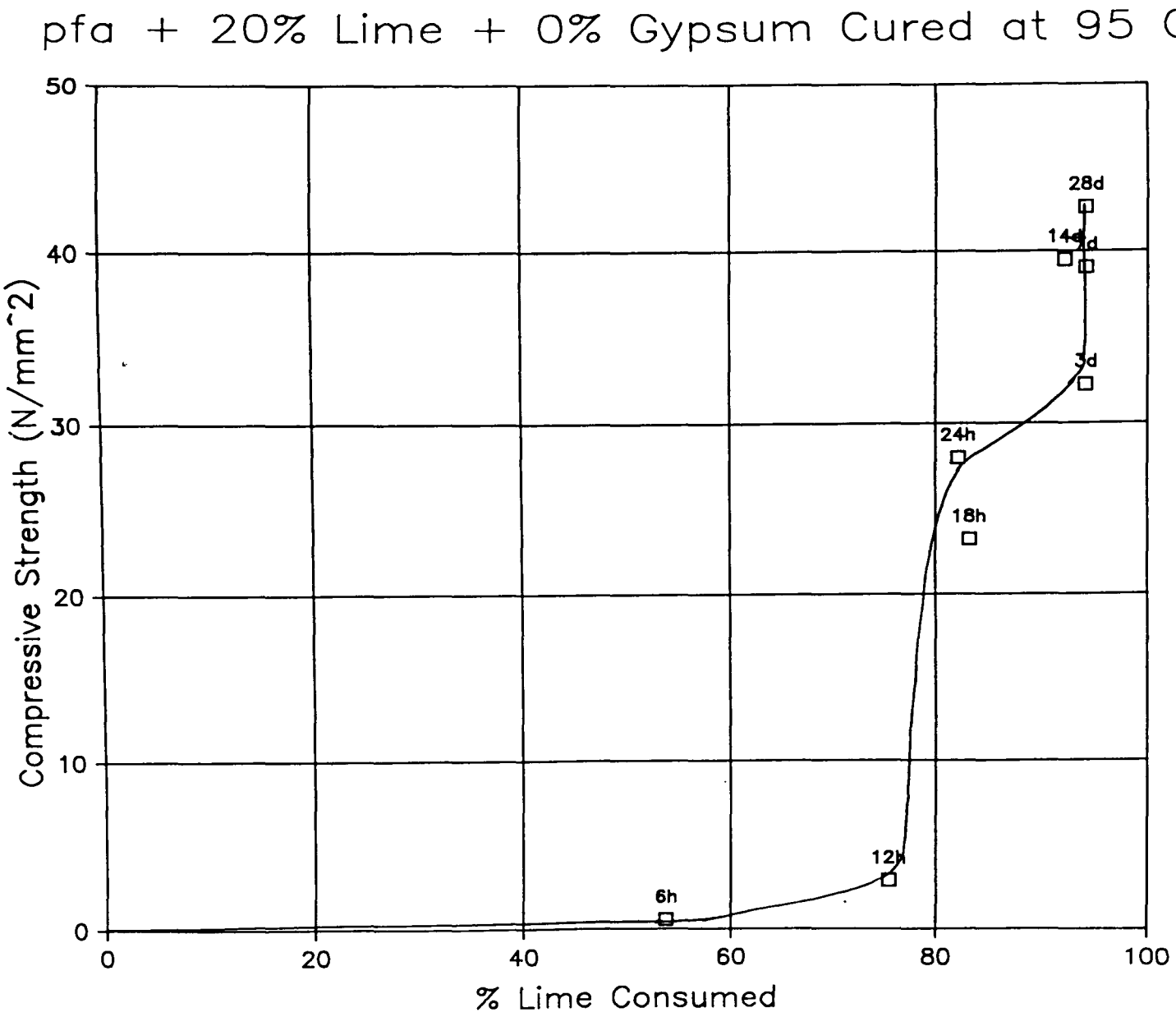


Figure 7.9b Compressive strength versus % lime consumption for system 1 cured up to 28 days. (curing time denoted, h = hours, d = days)

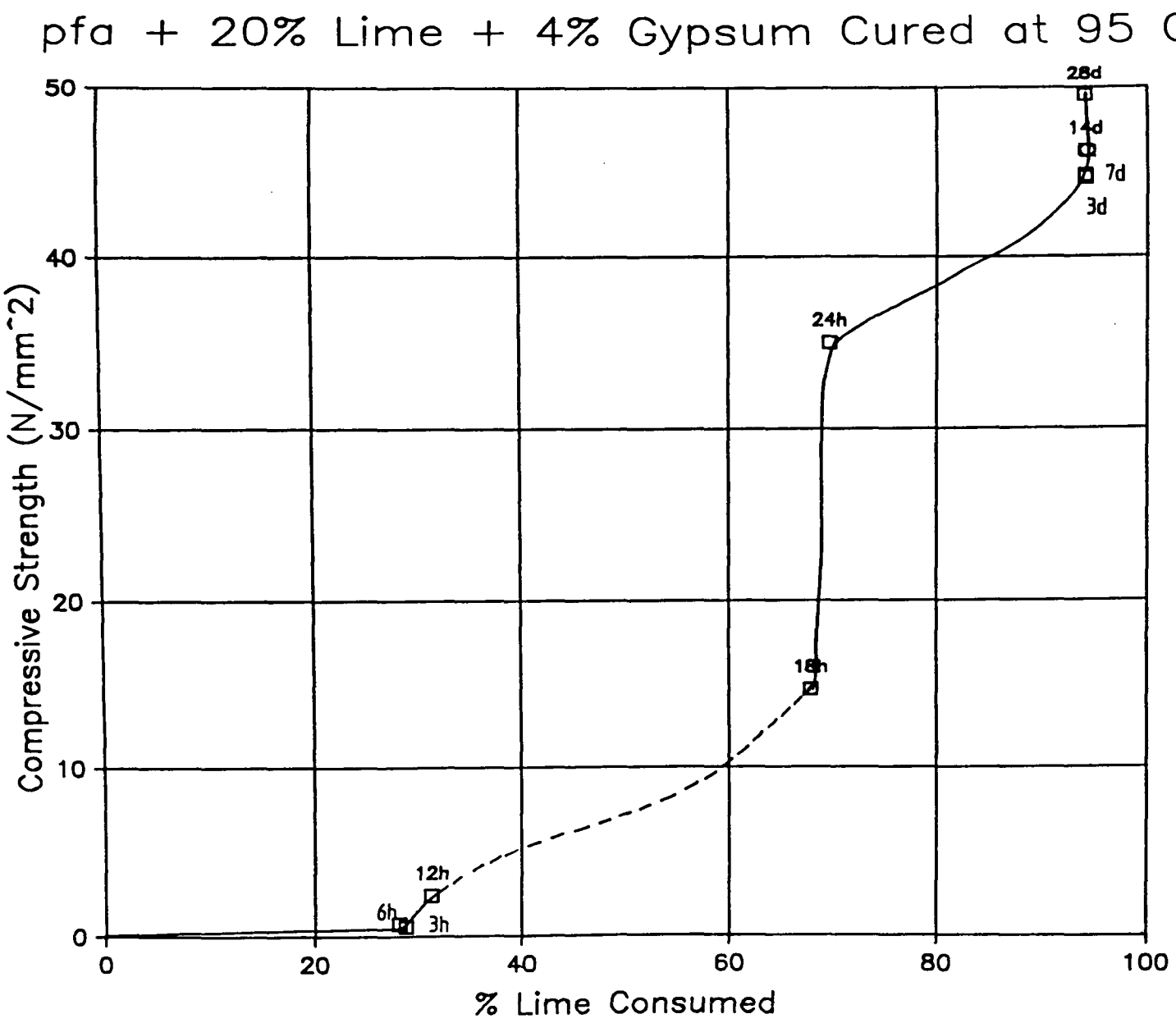


Figure 7.9c Compressive strength versus % lime consumption for system 2 cured up to 28 days. (curing time denoted, h = hours, d = days)

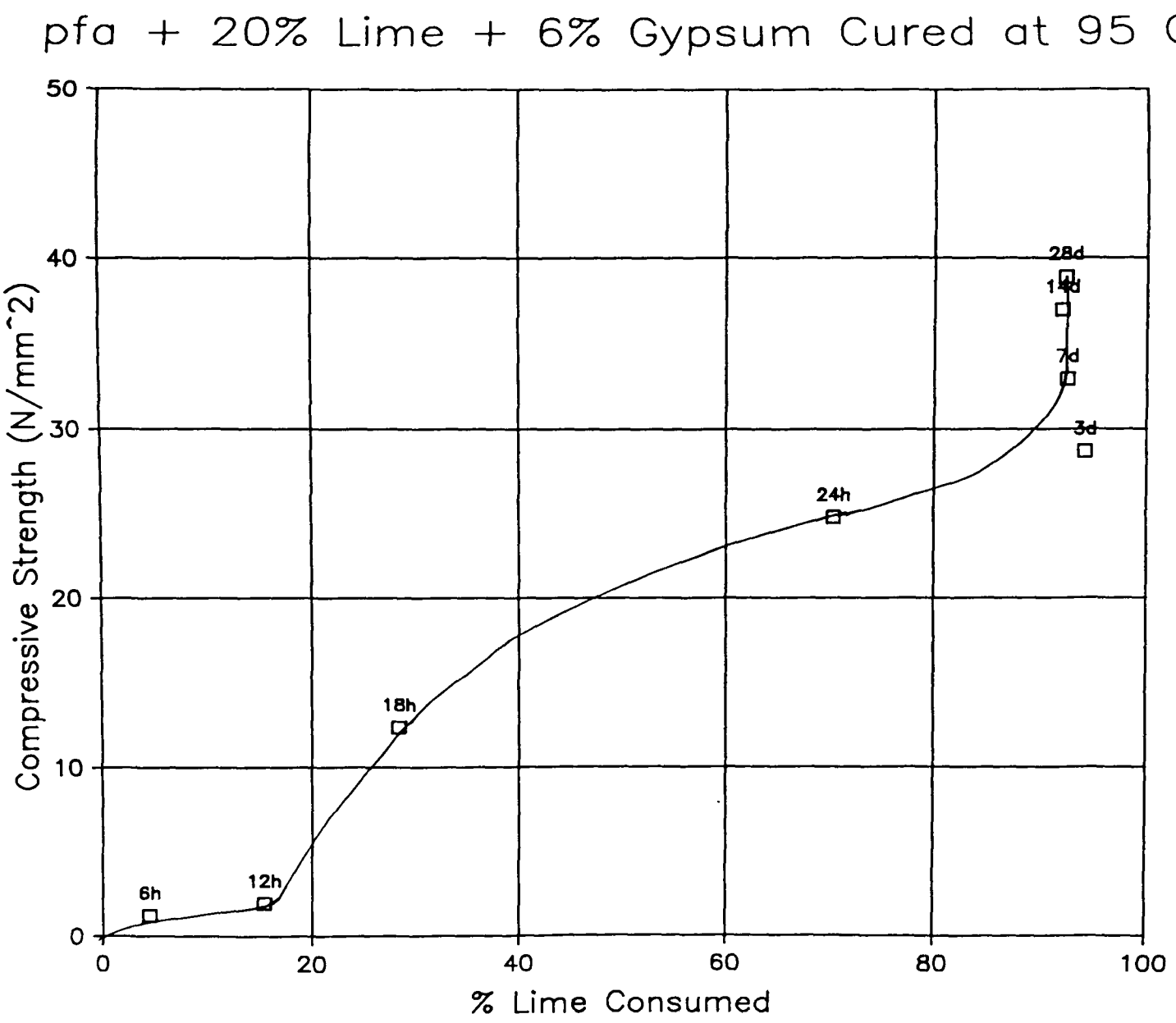


Figure 7.9d Compressive strength versus % lime consumption for system 3 cured up to 28 days.
(curing time denoted, h = hours, d = days)

CHAPTER EIGHT

TABLES, FIGURES AND PLATES

Table 8.1

Mass balance calculations derived from the DTG results for system D pfa.

A	B	C	D	E	F	G	H	I	J	K	L
3h	9.2614	.6158	.9318	8.3568	1.6714	.1613	.7705	.5702	.3241	3.8781	19.39
6h	10.1307	.5931	.5457	9.0591	1.8118	.1748	.3708	.2744	.7695	8.4940	42.47
12h	9.6670	.2104	.2904	8.6045	1.7209	.1661	.1243	.0920	1.2524	14.5551	72.78
18h	11.7560	.1809	.2925	10.4534	2.0907	.2018	.0908	.0672	1.6409	15.6974	78.49
24h	10.1905	.2069	.2046	9.0528	1.8106	.1747	.0299	.0221	1.4068	15.5404	77.70
3d	13.4345	.1943	.4627	11.9682	2.3936	.2310	.2317	.1715	1.7969	15.0139	75.07
7d	12.3436	.0000	.3614	10.9852	2.1970	.2120	.1493	.1105	1.8745	17.0640	85.32
14d	11.5235	.0000	.3506	10.2577	2.0515	.1980	.1526	.1129	1.7406	16.9692	84.85
28d	9.8425	.0000	.1745	8.7397	1.7479	.1687	.0059	.0043	1.5749	18.0203	90.10

Keys To Calculations,
A = Curing Time (h=hours,d=days)
B = Final Weight of the Sample at ≈ 860°C (mg)
C = Weight of Free Lime in the Sample Detected by TG (mg)
D = Weight of CaCO3 in the Sample Detected by TG (mg)
E = Weight of pfa in the Sample Calculated Using Equation 7 of Appendix 1
F = Weight of the Lime in the Original Sample (mg = E*(20/100))
G = Weight of CaCO3 in the Original Sample Calculated By Determining The Level Of The Carbonation In The Original Lime (mg)
H = Weight of CaCO3 Formed During Curing (mg = D-G)
I = Weight of Lime Carbonated During Curing (mg = H*0.74)
J = Weight of Lime Consumed During Curing (mg = F-(C+G+I))
K = Percentage of Lime Consumed Based on pfa ((J/E)*100)
L = Percentage of Lime Consumed ((K/20)*100)

Table 8.2

EDAX results from TEM studies of the fibrous gel formed in system D pfa cured up to 28 days.

Curing Time (days)	Mean Ca/Si	Mean Al/Si	Mean K/Si	Mean S/Si	Mean Na/Si	Mean Fe/Si	Number of Analyses
.5	1.340	.390	.120	.030	.130	.080	8
.75	1.190	.420	.200	.050	.110	.090	4
1	1.120	.400	.030	.040	.010	.080	7
3	1.000	.390	.120	.030	.070	.080	8
7	.800	.310	.090	.020	.060	.020	8
14	.880	.310	.070	.020	.040	.060	11
28	.830	.320	.080	.030	.060	.050	14
Average	.984	.352	.092	.029	.065	.062	60

Table 8.3

XRD data for system 2 specimens when (a) in their dry as-mixed condition, and (b) immediately after mixing with 30.2% (by the weight of the pfa) of distilled water at room temperature.

(a)			(b)		
d	I	C	d	I	C
			0.961	w	E
0.756	vvs	G	0.755	vs	G
			0.552	w	E
0.536	w	M	0.538	w	M
0.488	s	L	0.486	ms	L
0.427	m	Q+G	0.426	m	Q+G
			0.386	w	E (+G?)
0.378	s	G	0.379	m	G
0.342	m	M	0.342	m	M
0.339	m	M	0.338	m	M
0.334	s	Q	0.335	s	Q
0.310	m	L	0.309	ms	L
0.309	m	G	0.304	m	G
0.302	w	Ca	0.302	m	Ca
0.288	w	M+G	0.288	mw	M+G
			0.277	w	E
0.268	w	M+Ha+G	0.268	w	M+Ha+G
0.261	vs	L	0.261	s	L
0.254	w	M+Mg	0.254	w	M+Mg
0.251	w	Ha+Ca	0.251	w	Ha+Ca

Keys : d - d spacings (nm), I - peak intensity, C - compound, L - lime, Ca - calcite, E - ettringite, G - gypsum, M - mullite, Q - quartz, Mg - magnitite, Ha - haematite.

Table 8.4

Mass balance calculations derived from the DTG results for system 2 when in the dry as-mixed condition and after mixing with 30.2% of distilled water.

a) Mass Balance Calculations For The Lime Consumed; Keys to Calculations Are Similar To Table 8.5

	A	B	C	D	E	F	G	H	I	J	K	L
Dry (No Curing)		11.6862	1.891	.121	10.0830	2.017	.116	.005	.004	.006	.059	.293
Moist (No Curing)		10.1576	1.215	.667	8.7641	1.753	.101	.566	.419	.018	.206	1.029

b) Mass Balance Calculations For The Gypsum Consumed

	1	2	3	4	5	6	7	8	9	10	11
Dry (No Curing)		11.6862	.406	10.0830	.000	.403	-.003	-.027	4.027	-.664	100.664
Moist (No Curing)		10.1576	.603	8.7641	.117	.351	-.135	-1.544	6.884	-28.922	128.922

Keys To Calculations,
1 = Condition of sample
2 = Final weight of the sample (mg)
3 = Weight of free gypsum in the sample as detected by DTG ≈130-160 degrees C (mg)
4 = Weight of pfa in the samples calculated using Equation 9 of Appendix 1
5 = Weight of gypsum present in the original pfa (mg=1.34% x D)
6 = Weight of gypsum added to the original sample (mg = 4% x D)
7 = Weight of gypsum consumed (mg = E+F-C)
8 = Percentage of gypsum consumed based on the weight of the original pfa ((G/D)x100)
9 = Percentage of unconsumed gypsum based on the weight of the original pfa ((C/D)x100)
10 = Percentage of gypsum consumed ({H/[4+1.34]} x 100)
11 = Percentage of gypsum unconsumed ({I/[4+1.34]} x 100)

c) Mass Balance Calculations For The Ettringite Present

	i	ii	iii	iv	v	vi
Dry (No Curing)		11.6862	10.0830	-	-	-
Moist (No Curing)		10.1576	8.7641	.1940	.5201	5.9300

Keys to Calculations,
i = Condition of sample
ii = Final weight of the sample (mg)
iii = Weight of the pfa in the sample calculated using Equation 9 of Appendix 1.
iv = Weight loss detected by TG at 92°C (mg)
v = Weight of ettringite present assuming the weight loss in column iv is solely due to the dehydration of ettringite losing 26 H2O (iv/0.373 mg)
vi = Percentage of ettringite present based on the weight of the pfa ((v/iii)*100)

Table 8.4

Mass balance calculations derived from the DTG results for system 2 when in the dry as-mixed condition and after mixing with 30.2% of distilled water.

a) Mass Balance Calculations For The Lime Consumed; Keys to Calculations Are Similar To Table 8.5

	A	B	C	D	E	F	G	H	I	J	K	L
Dry (No Curing)		11.6862	1.891	.121	10.0830	2.017	.116	.005	.004	.006	.059	.293
Moist (No Curing)		10.1576	1.215	.667	8.7641	1.753	.101	.566	.419	.018	.206	1.029

b) Mass Balance Calculations For The Gypsum Consumed

	1	2	3	4	5	6	7	8	9	10	11
Dry (No Curing)		11.6862	.406	10.0830	.000	.403	-.003	-.027	4.027	-.664	100.664
Moist (No Curing)		10.1576	.603	8.7641	.117	.351	-.135	-1.544	6.884	-28.922	128.922

Keys To Calculations,
1 = Condition of sample
2 = Final weight of the sample (mg)
3 = Weight of free gypsum in the sample as detected by DTG ≈130-160 degrees C (mg)
4 = Weight of pfa in the samples calculated using Equation 9 of Appendix 1
5 = Weight of gypsum present in the original pfa (mg=1.34% x D)
6 = Weight of gypsum added to the original sample (mg = 4% x D)
7 = Weight of gypsum consumed (mg = E+F-C)
8 = Percentage of gypsum consumed based on the weight of the original pfa ((G/D)x100)
9 = Percentage of unconsumed gypsum based on the weight of the original pfa ((C/D)x100)
10 = Percentage of gypsum consumed ({H/[4+1.34]} x 100)
11 = Percentage of gypsum unconsumed ({I/[4+1.34]} x 100)

c) Mass Balance Calculations For The Ettringite Present

	i	ii	iii	iv	v	vi
Dry (No Curing)		11.6862	10.0830	-	-	-
Moist (No Curing)		10.1576	8.7641	.1940	.5201	5.9300

Keys to Calculations,
i = Condition of sample
ii = Final weight of the sample (mg)
iii = Weight of the pfa in the sample calculated using Equation 9 of Appendix 1.
iv = Weight loss detected by TG at 92°C (mg)
v = Weight of ettringite present assuming the weight loss in column iv is solely due to the dehydration of ettringite losing 26 H2O (iv/0.373 mg)
vi = Percentage of ettringite present based on the weight of the pfa ((v/iii)x100)

Table 8.5

Mass balance calculations derived from the DTG results for system 2.

A	B	C	D	E	F	G	H	I	J	K	L
3h	11.0802	1.15	.2522	9.5805	1.9161	.1130	.1392	.1030	.5521	5.7625	28.81
6h	10.8743	1.18	.1909	9.3929	1.8786	.1108	.0801	.0592	.5295	5.6372	28.19
12h	9.0339	.91	.1822	7.8072	1.5614	.0921	.0901	.0667	.4892	6.2654	31.33
18h	10.7438	.39	.2416	9.2891	1.8578	.1096	.1320	.0977	1.2612	13.5776	67.89
24h	9.6043	.32	.2118	8.3032	1.6606	.0980	.1138	.0842	1.1579	13.9456	69.73
3d	8.9942	.00	.0000	7.7423	1.5485	.0914	.0000	.0000	1.4571	18.8200	94.10
7d	8.3643	.00	.0000	7.2001	1.4400	.0850	.0000	.0000	1.3551	18.8200	94.10
14d	8.8045	.00	.0000	7.5789	1.5158	.0894	.0000	.0000	1.4264	18.8200	94.10
28d	8.5367	.00	.0000	7.3485	1.4697	.0867	.0000	.0000	1.3830	18.8200	94.10

Keys To Calculations,
A = Curing Time (h=hours,d=days)
B = Final Weight of the Sample at ≈ 860°C (mg)
C = Weight of Free Lime in the Sample Detected by TG (mg)
D = Weight of CaCO3 in the Sample Detected by TG (mg)
E = Weight of pfa in the Sample Calculated Using Equation 9 of Appendix 1
F = Weight of the Lime in the Original Sample (mg = E*(20/100))
G = Weight of CaCO3 in the Original Sample Calculated By Determining The Level Of The Carbonation In The Original Lime (mg)
H = Weight of CaCO3 Formed During Curing (mg = D-G)
I = Weight of Lime Carbonated During Curing (mg = H*0.74)
J = Weight of Lime Consumed During Curing (mg = F-{C+G+I})
K = Percentage of Lime Consumed Based on pfa ({J/E}*100)
L = Percentage of Lime Consumed ({K/20}*100)

Table 8.6

EDAX results from TEM studies of the fibrous gel formed in system 2 cured up to 28 days.

Curing Time (days)	Mean Ca/Si	Mean Al/Si	Mean K/Si	Mean S/Si	Mean Na/Si	Mean Fe/Si	Number of Analyses
.5	Insufficient Fibrous Gel to Obtain a Representative Analyses						
.75	.794	.293	.057	.059	-	.030	8
1	.897	.478	.046	.100	-	.044	5
3	1.001	.362	.055	.066	-	.040	9
7	.839	.451	.091	.091	-	.059	25
14	1.081	.412	.111	.110	.041	.146	19
28	1.075	.467	.124	.089	.349	.142	21
Average	.965	.424	.094	.090	.195	.092	87
Ash Only	.049	.781	.112	.014	.158	.045	

Table 8.7

Mass balance calculations derived from the DTG results for system 3.

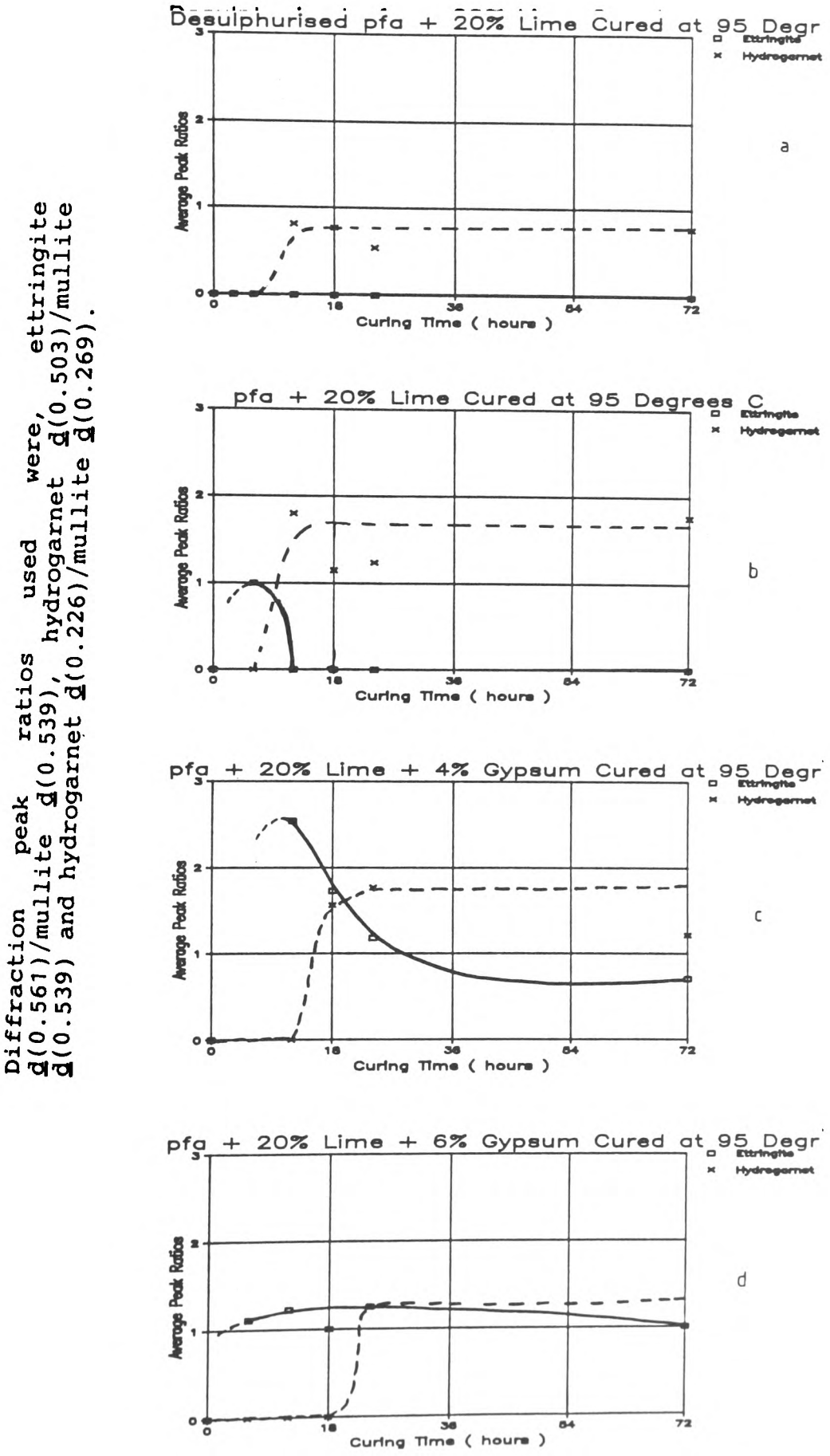
A	B	C	D	E	F	G	H	I	J	K	L
3h	10.9671	.9110	1.1950	9.5128	1.9025	.1084	1.0875	.8047	.0784	.8241	4.12
6h	10.9451	.8890	1.2110	9.4968	1.8994	.1083	1.1027	.8160	.0861	.9063	4.53
12h	9.6820	.6320	1.0300	8.3940	1.6788	.0957	.9343	.6914	.2597	3.0941	15.47
18h	9.3616	.4250	.9620	8.1105	1.6221	.0925	.8695	.6435	.4612	5.6862	28.43
24h	10.4532	.2210	.3800	8.9407	1.7881	.1019	.2781	.2058	1.2594	14.0866	70.43
3d	9.3578	.0000	.0743	7.9595	1.5919	.0907	.0000	.0000	1.5012	18.8600	94.30
7d	11.1990	.0000	.1530	9.5363	1.9073	.1087	.0443	.0328	1.7658	18.5163	92.58
14d	10.6019	.0000	.1580	9.0300	1.8060	.1029	.0551	.0407	1.6623	18.4088	92.04
28d	10.2141	.0000	.1440	8.6984	1.7397	.0992	.0448	.0332	1.6073	18.4785	92.39

Keys To Calculations,
A = Curing Time (h=hours,d=days)
B = Final Weight of the Sample at ≈ 860°C (mg)
C = Weight of Free Lime in the Sample Detected by TG (mg)
D = Weight of CaCO3 in the Sample Detected by TG (mg)
E = Weight of pfa in the Sample Calculated Using Equation 9 of Appendix 1
F = Weight of the Lime in the Original Sample (mg = E*(20/100))
G = Weight of CaCO3 in the Original Sample Calculated By Determining The Level Of The Carbonation In The Original Lime (mg)
H = Weight of CaCO3 Formed During Curing (mg = D-G)
I = Weight of Lime Carbonated During Curing (mg = H*0.74)
J = Weight of Lime Consumed During Curing (mg = F-{C+G+I})
K = Percentage of Lime Consumed Based on pfa ({J/E}*100)
L = Percentage of Lime Consumed ({K/20}*100)

Table 8.8

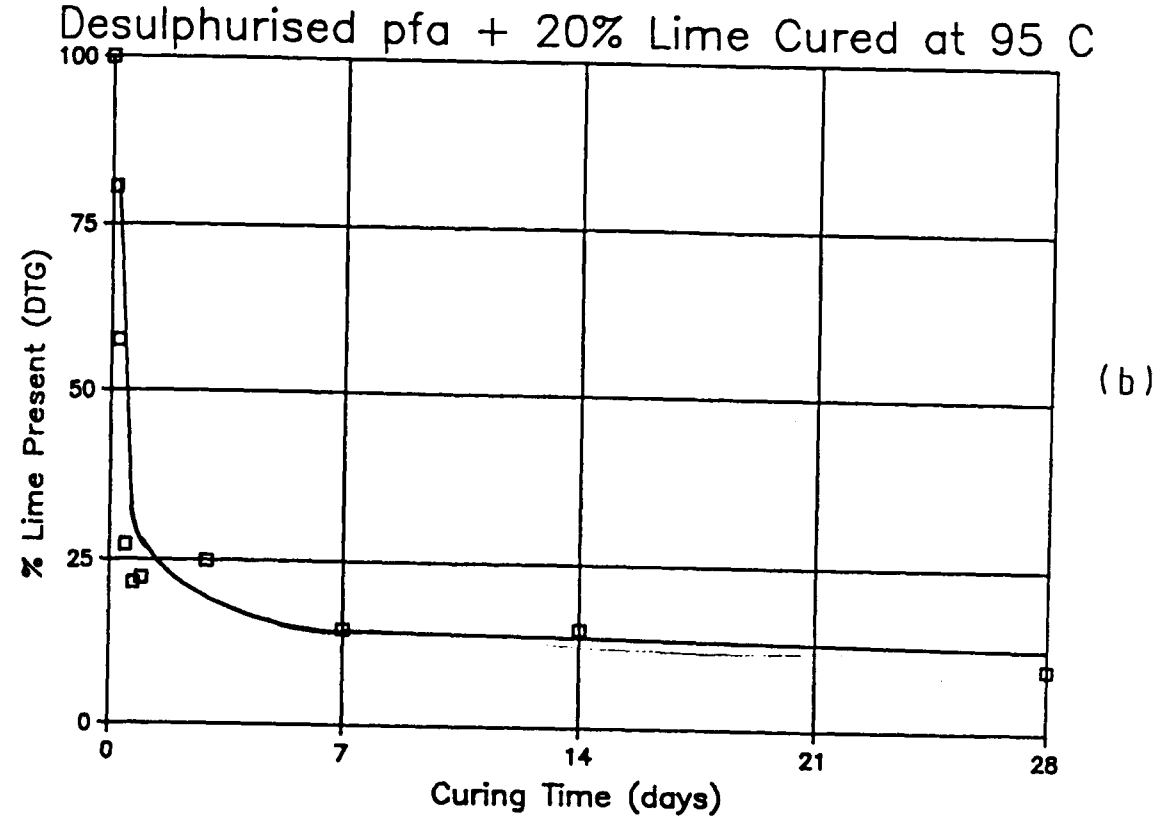
EDAX results from TEM studies of the fibrous gel formed in system 3 cured up to 28 days.

Curing Time (days)	Mean Ca/Si	Mean Al/Si	Mean K/Si	Mean S/Si	Mean Na/Si	Mean Fe/Si	Number of Analyses
.5	Insufficient Fibrous Gel to Obtain Representative Analyses						
.75	.893	.606	.126	.067	.044	.078	3
1	.780	.402	.077	.052	.025	.055	8
3	.635	.394	.190	.098	.034	.046	9
7	.630	.380	.074	.042	.040	.049	11
14	.627	.379	.074	.042	.039	.049	8
28	.553	.446	.081	.041	.041	.059	9



Diffraction peak ratios used were, ettringite d(0.561)/mullite d(0.539), hydrogarnet d(0.503)/mullite d(0.539) and hydrogarnet d(0.226)/mullite d(0.269).

Figure 8.1 Relative diffraction peak height ratios for ettringite and hydrogarnet plotted against curing time (up to 3 days) for a) system D pfa, b) system 1, c) system 2, and d) system 3.



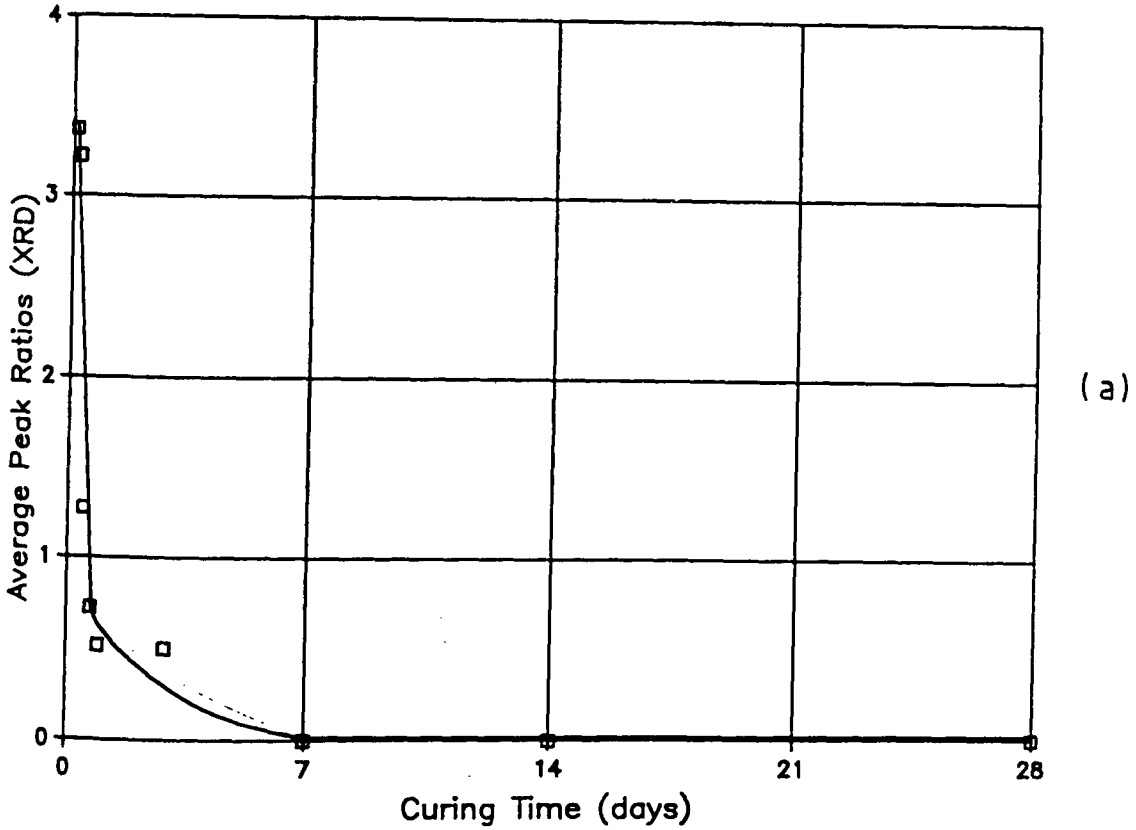


Figure 8.2 a) Relative diffraction peak height ratios for lime plotted against curing time (up to 28 days) for system D pfa. Diffraction peaks used were lime d(0.490)/quartz d(0.426) and lime d(0.262)/mullite d(0.269).

b) Percentage of residual lime (determined from DTG analysis) plotted against curing time (up to 28 days) for system D pfa.

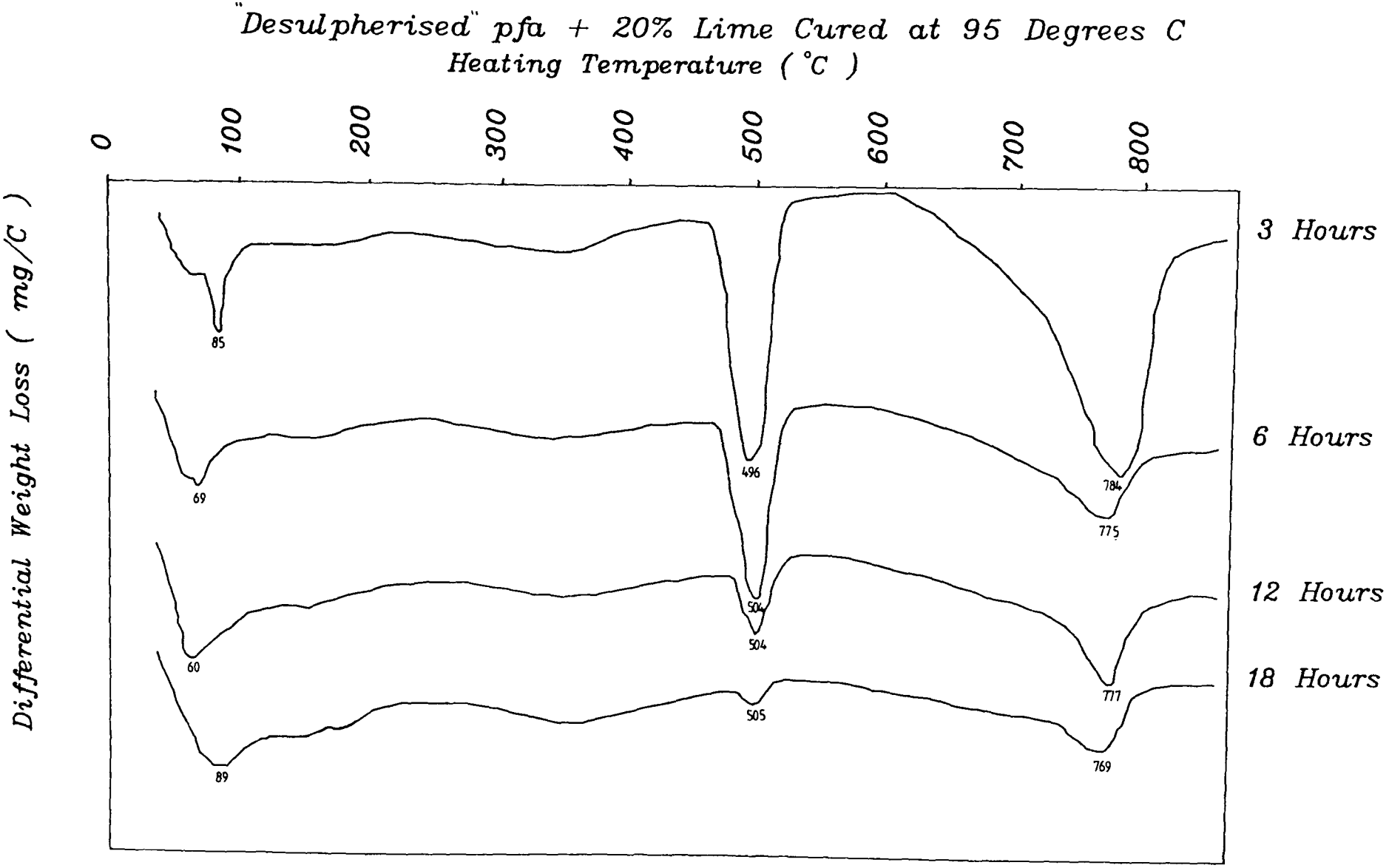


Figure 8.3 DTG thermograms for system D pfa at curing times from 3 hours to 18 hours.

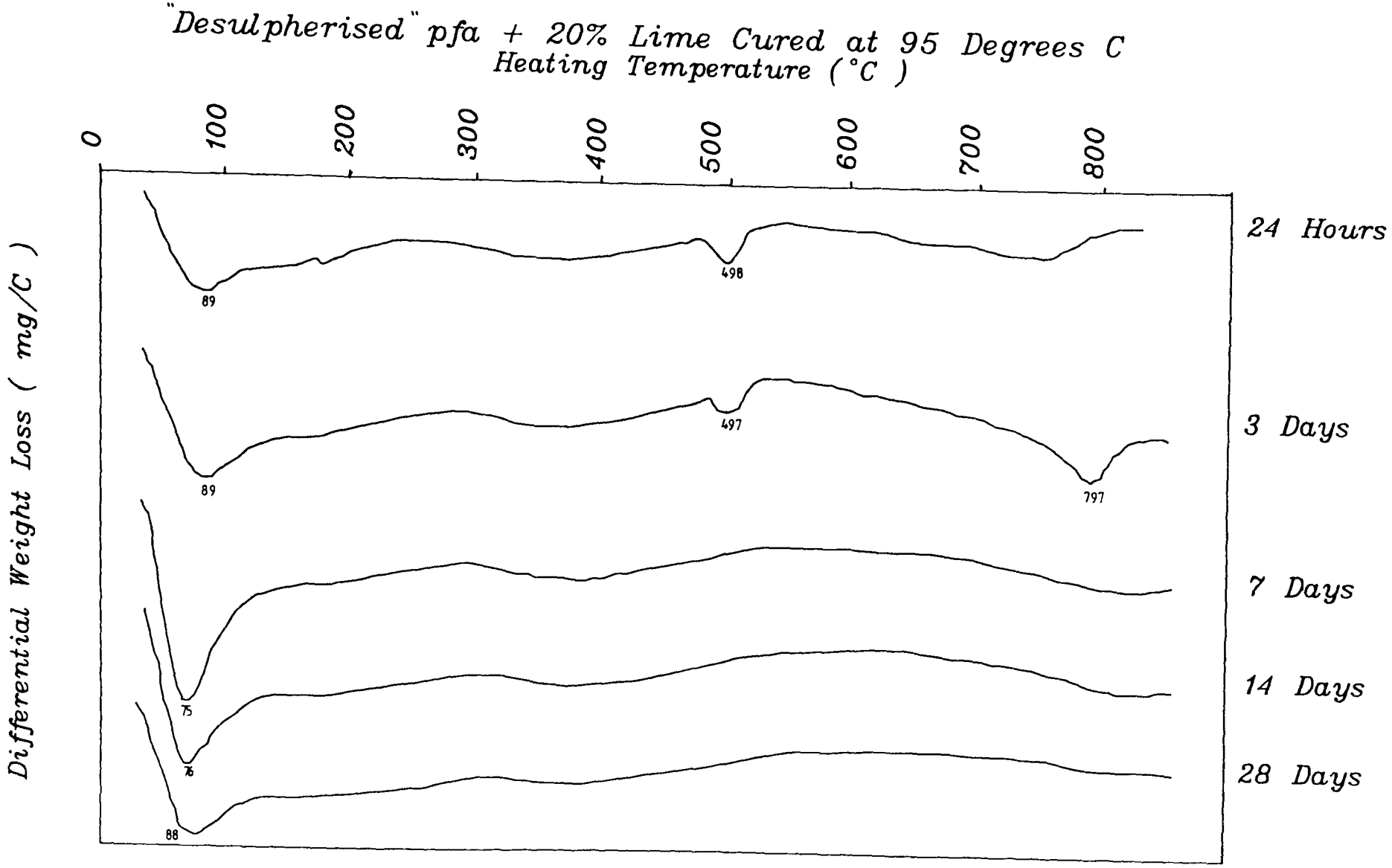


Figure 8.4 DTG thermograms for system D pfa at curing times from 24 hours to 28 days.

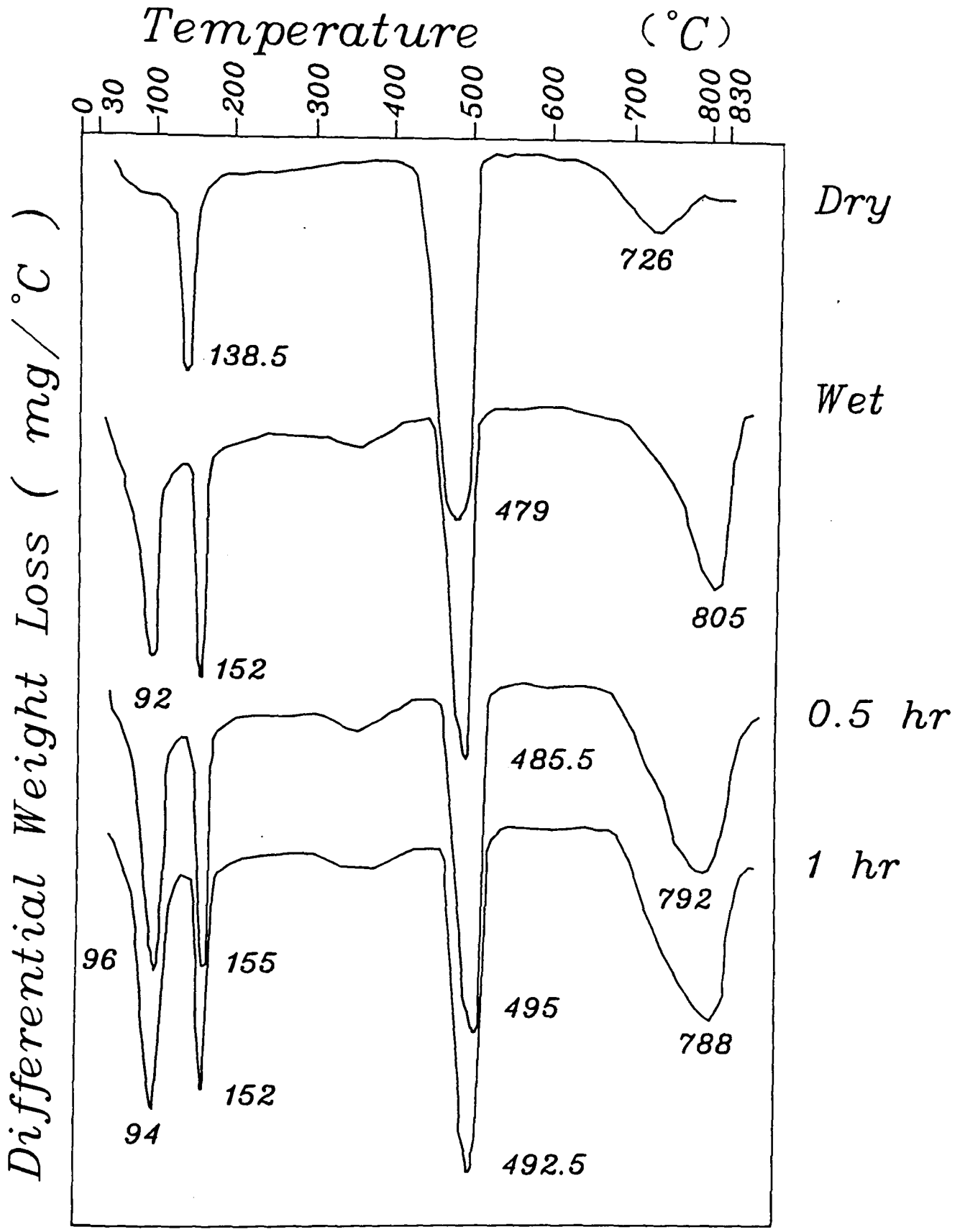


Figure 8.5 DTG thermograms for system 2 specimens in their dry as-mixed condition, after mixing with 30.2% of distilled water, and when cured at 95°C and 100% r.h. for 0.5 and 1 hour.

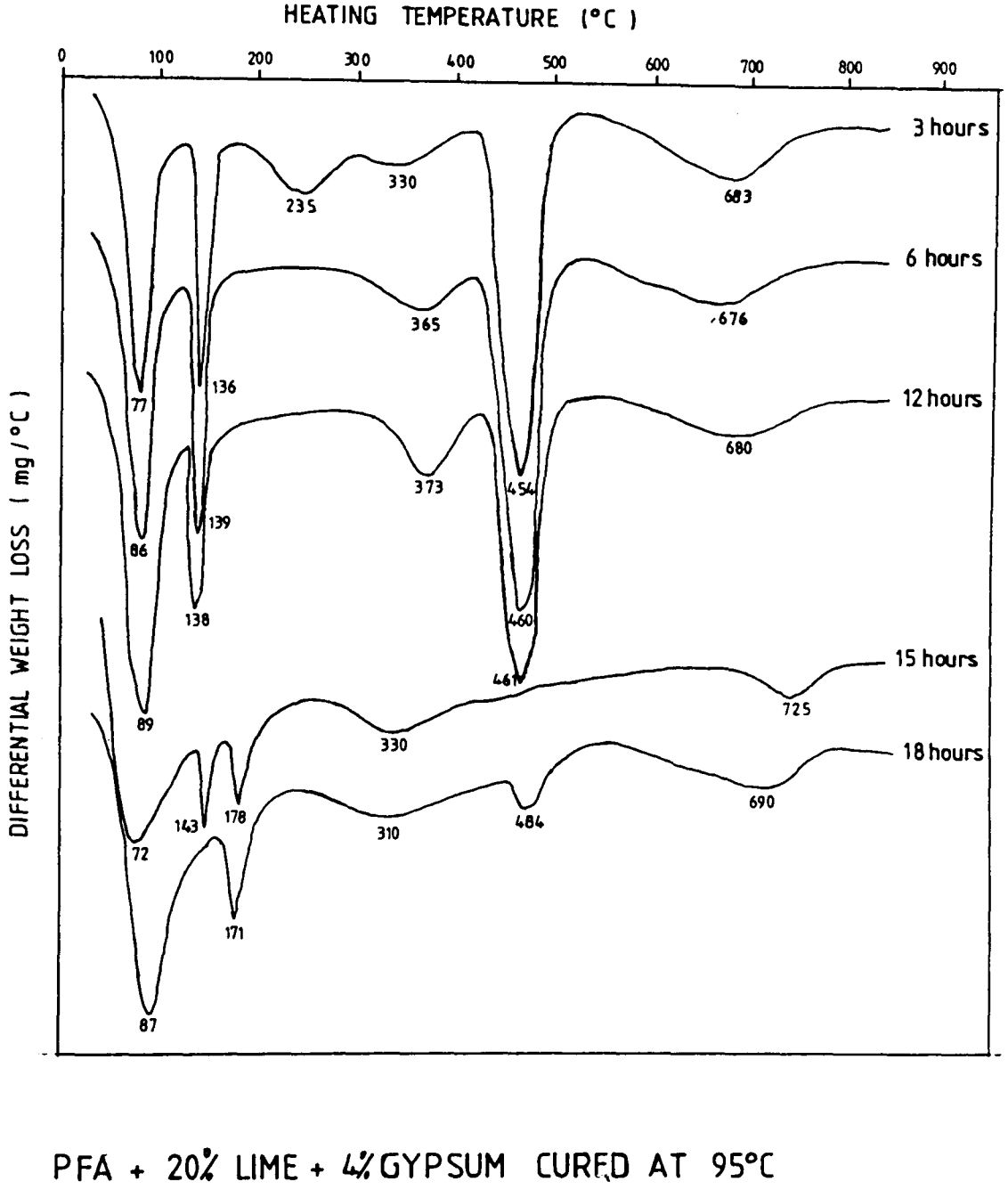


Figure 8.6 DTG thermograms for system 2 at curing times from 3 hours to 18 hours.

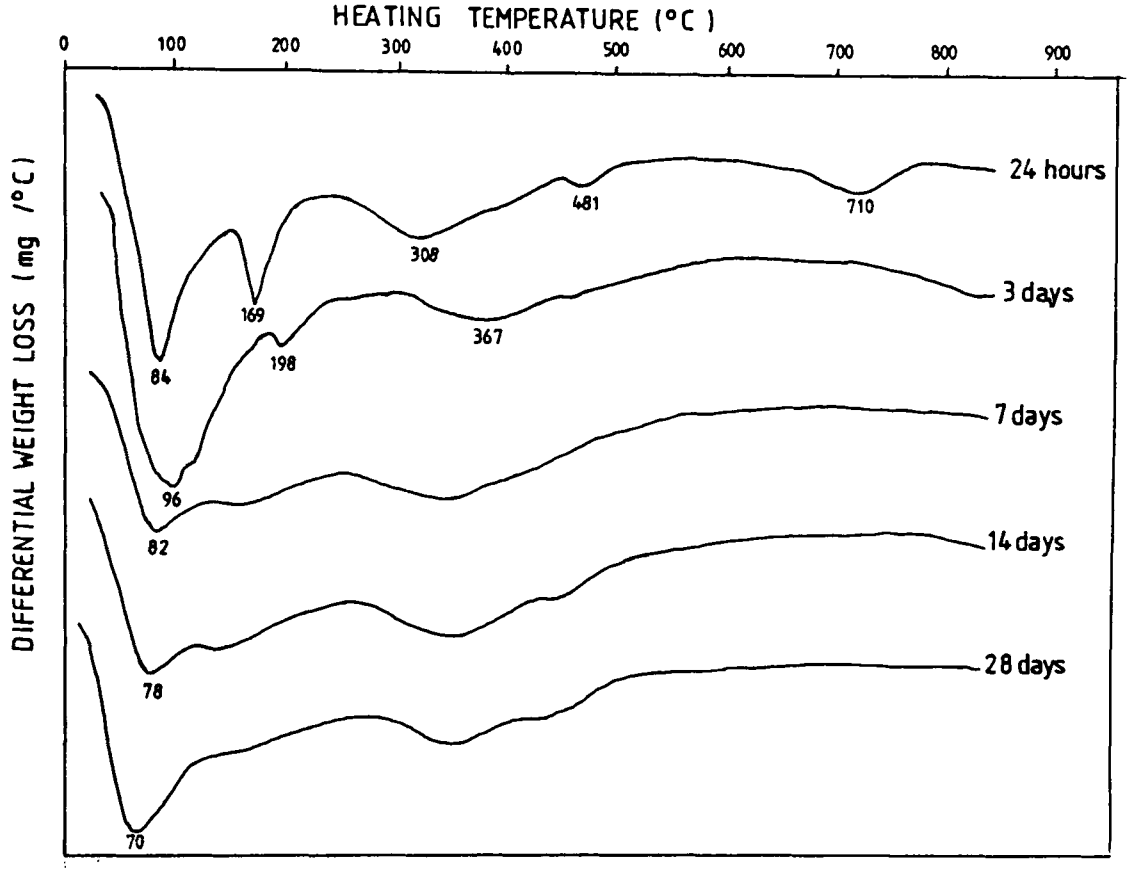


Figure 8.7 DTG thermograms for system 2 at curing times from 24 hours to 28 days.

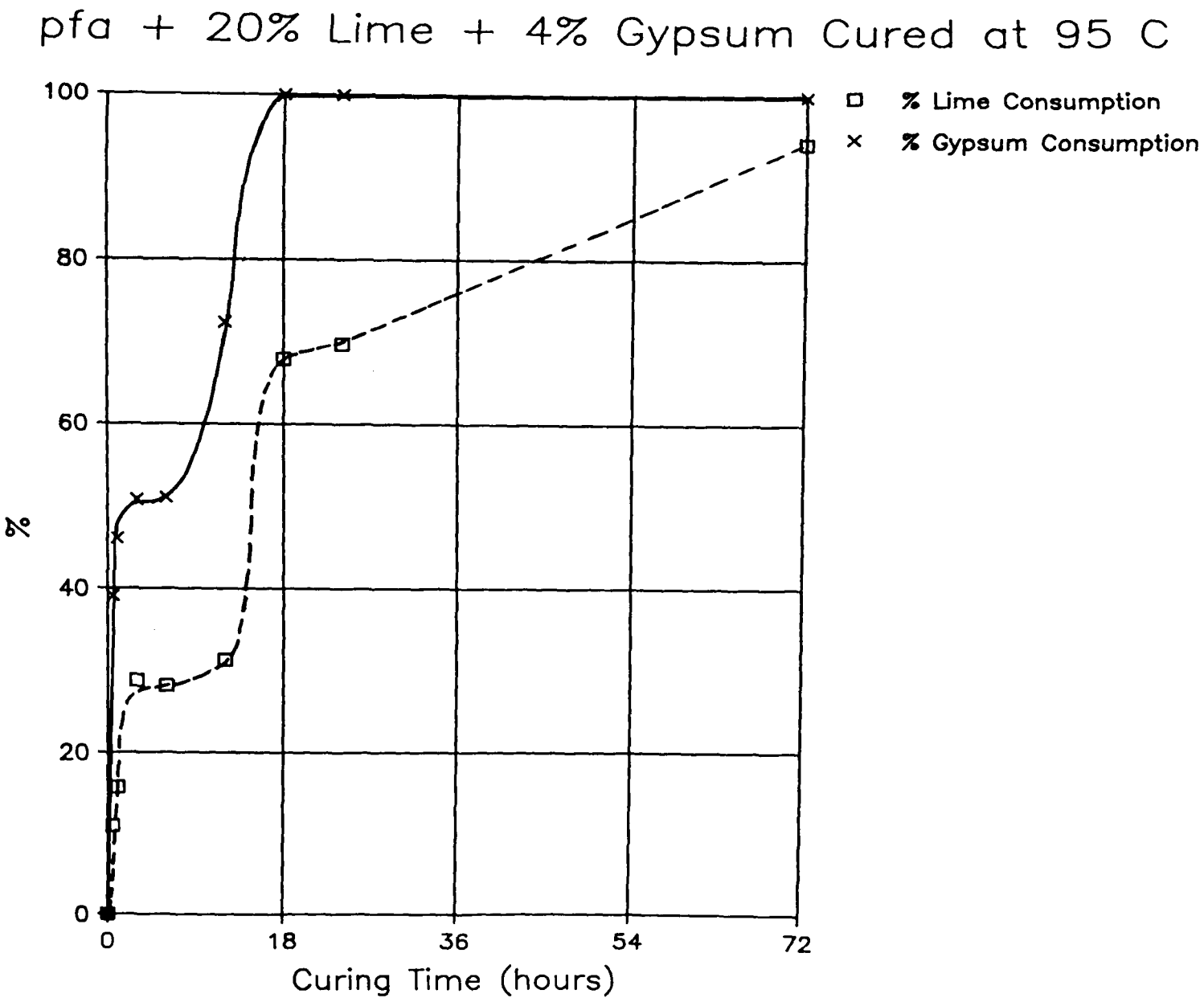


Figure 8.8 Percentage lime and gypsum consumption versus curing time (up to 3 days) derived from the DTG data.

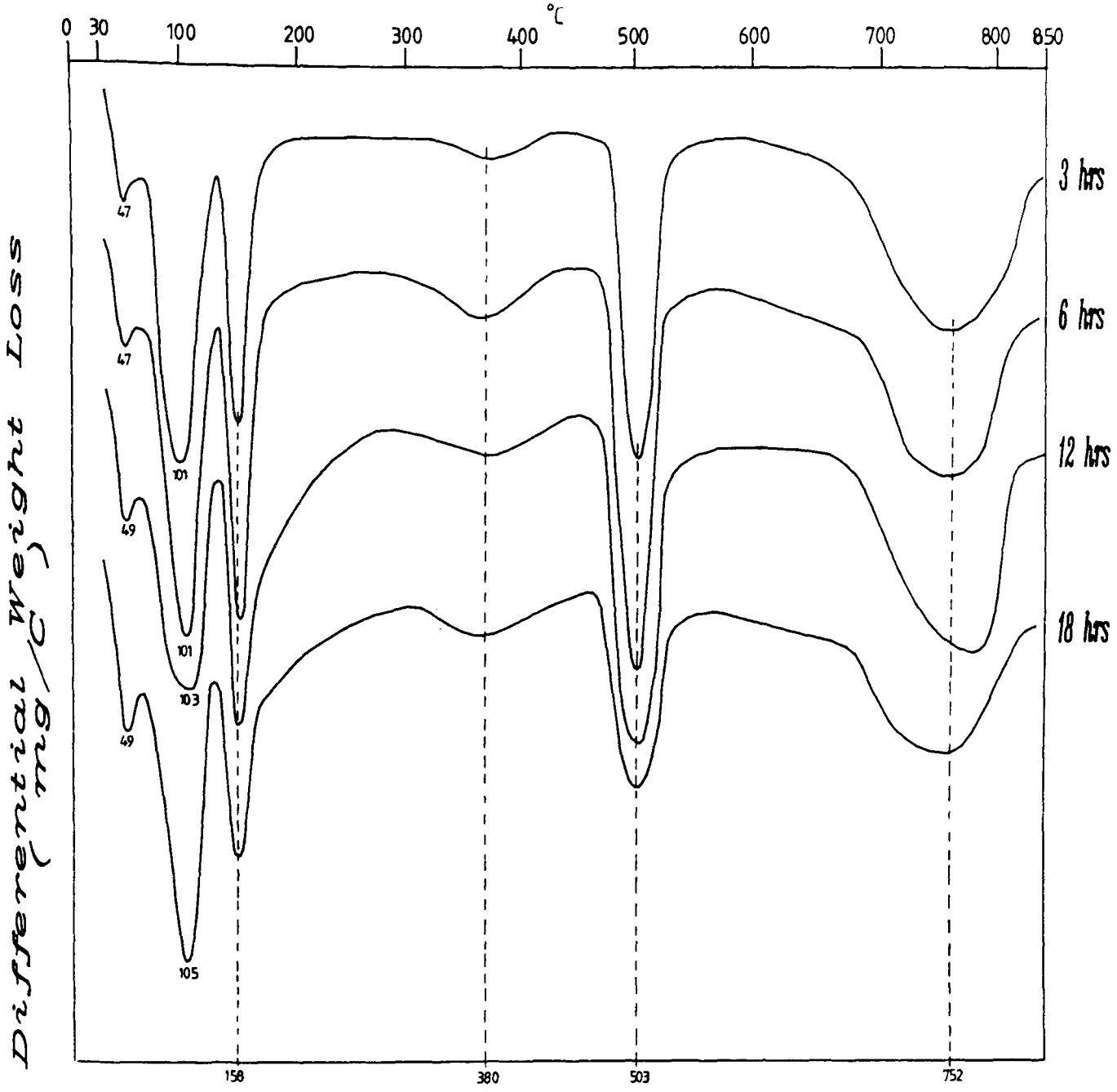


Figure 8.9 DTG thermograms for system 3 at curing times from 3 hours to 18 hours.

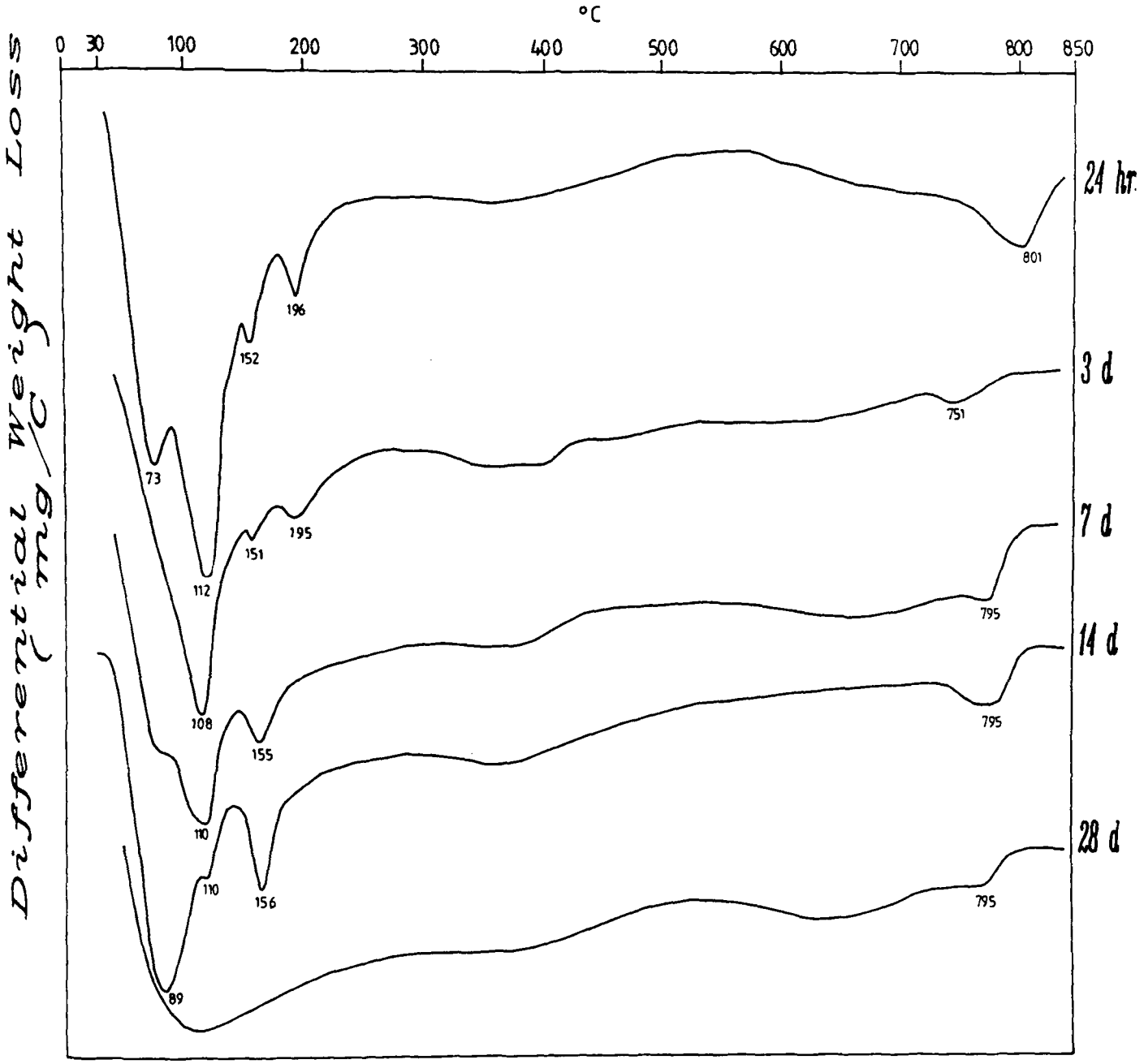


Figure 8.10 DTG thermograms for system 3 at curing times from 24 hours to 28 days.

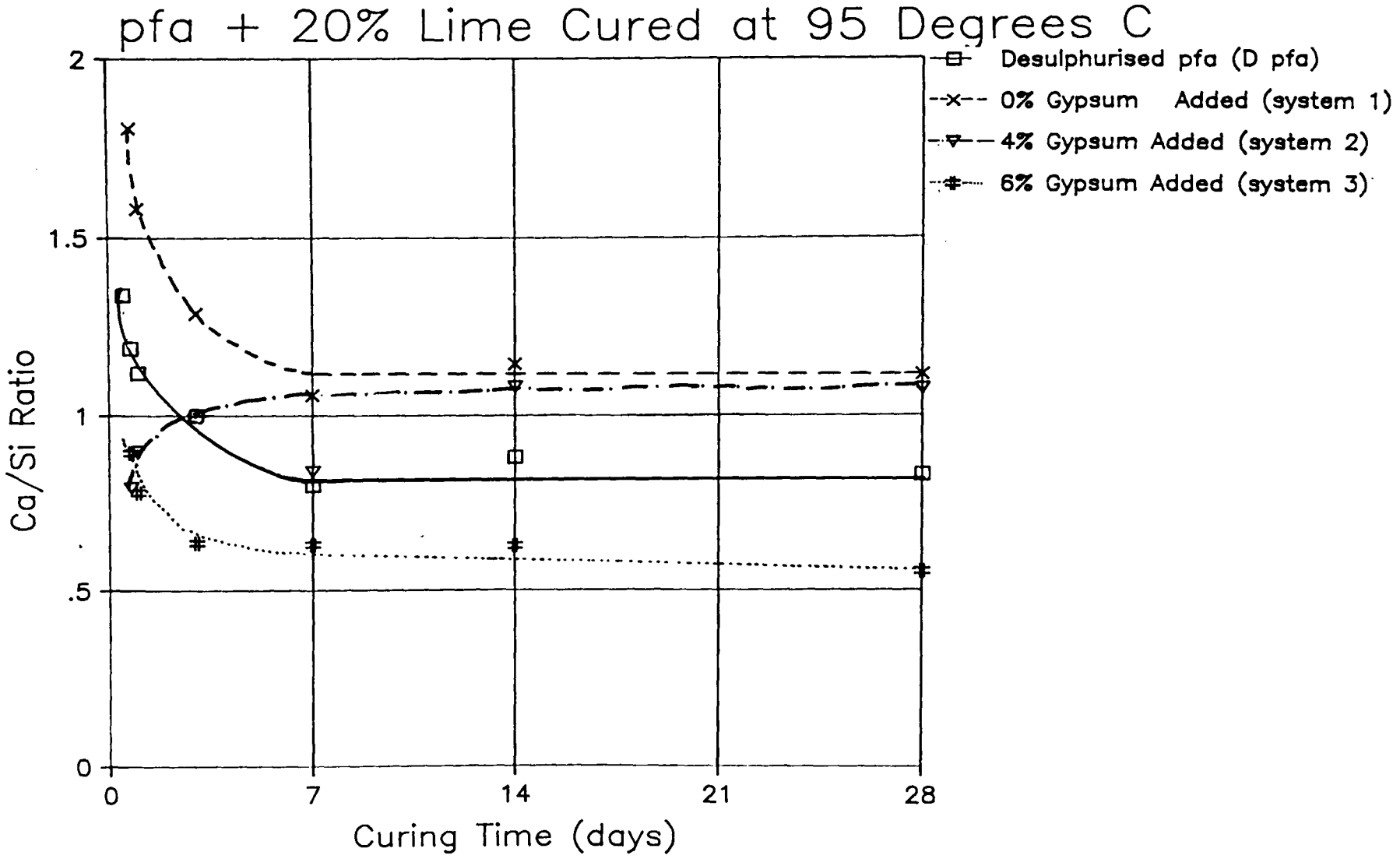


Figure 8.11 Ca/Si ratio versus curing time (up to 28 days) for system D pfa, system 1, system 2 and system 3.

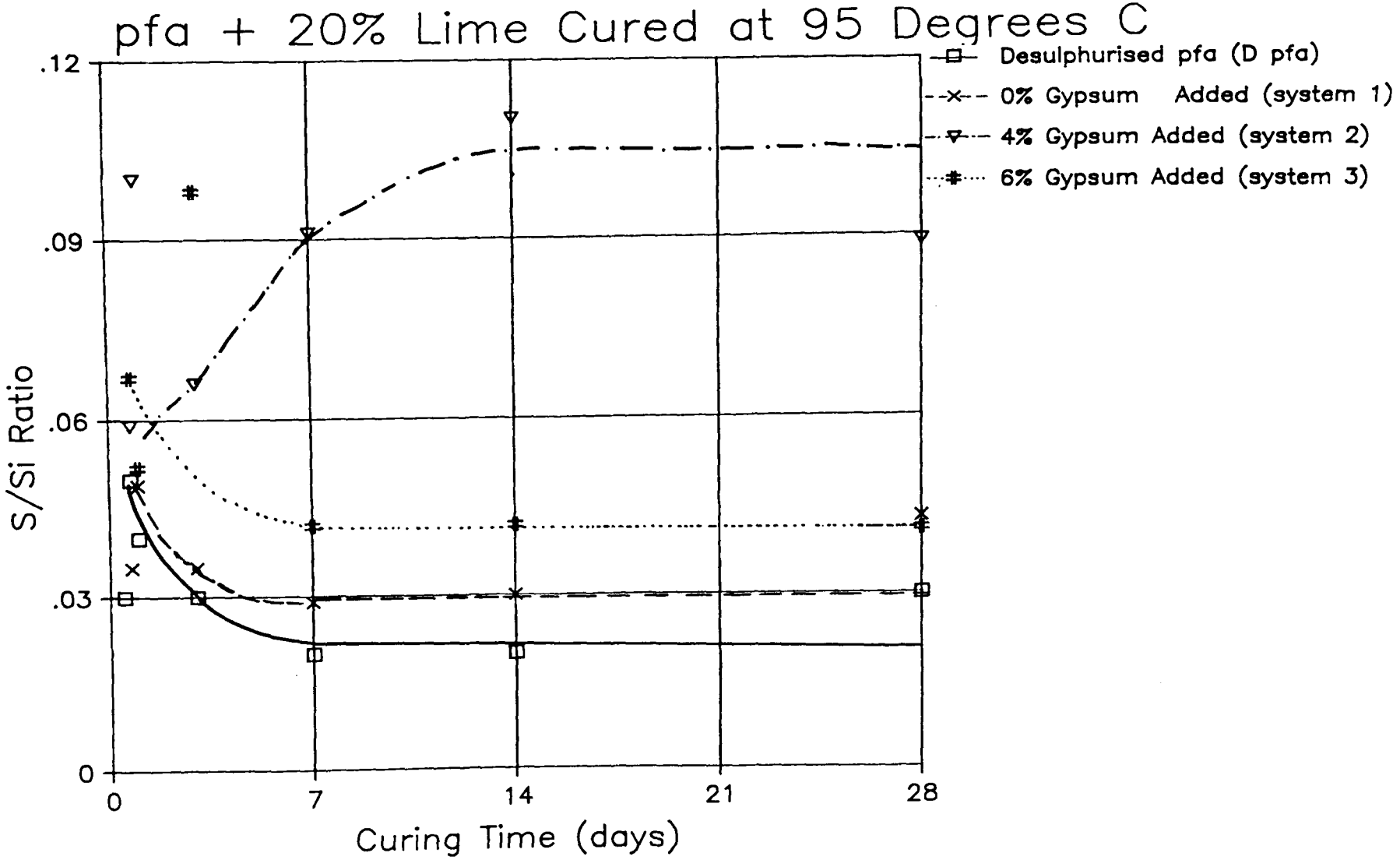


Figure 8.12 S/Si ratio versus curing time (up to 28 days) for system D pfa, system 1, system 2 and system 3.

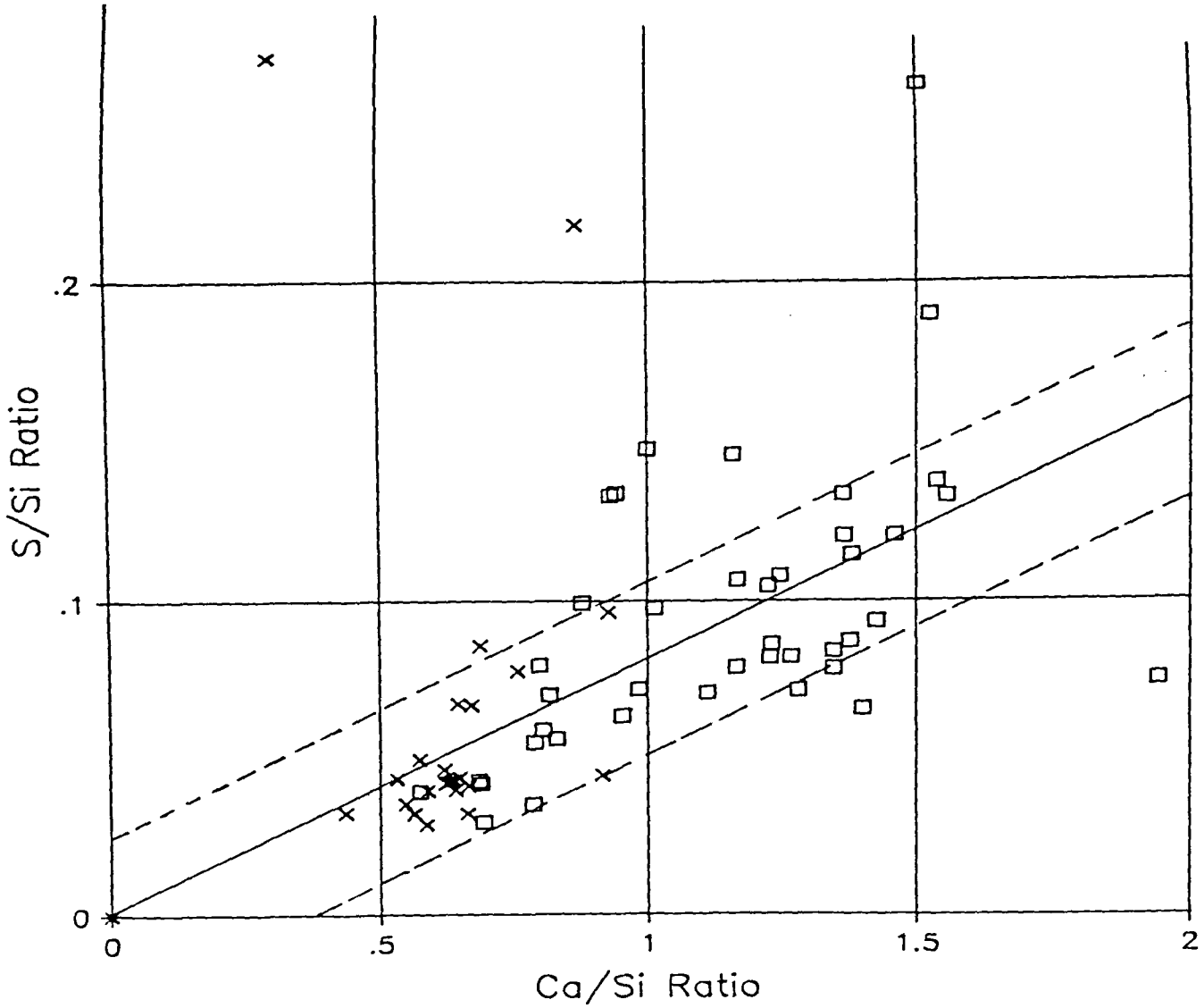


Figure 8.13 S/Si ratio versus Ca/Si for fibrous gel formed in system 2 and system 3.

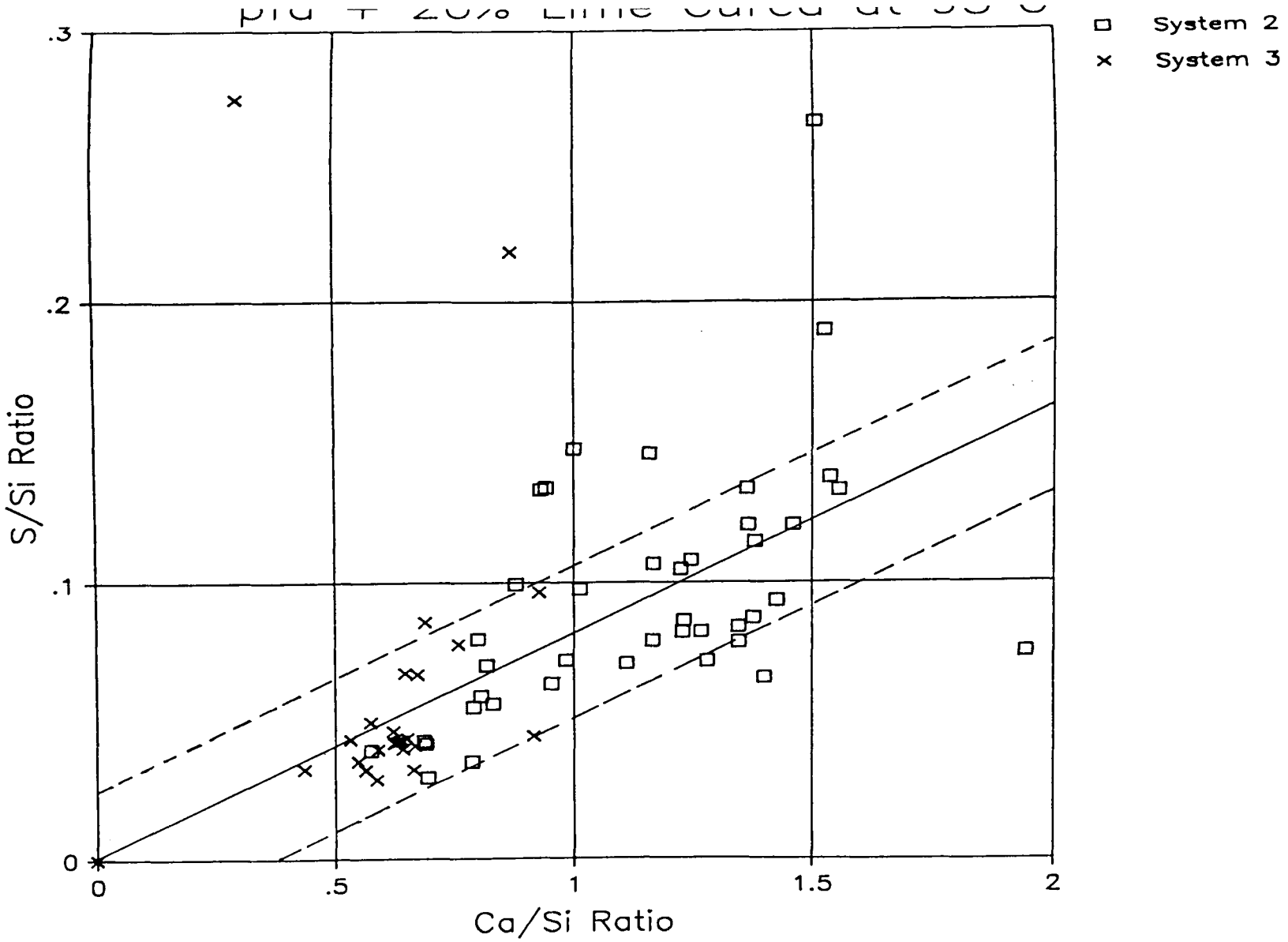


Figure 8.13 S/Si ratio versus Ca/Si for fibrous gel formed in system 2 and system 3.

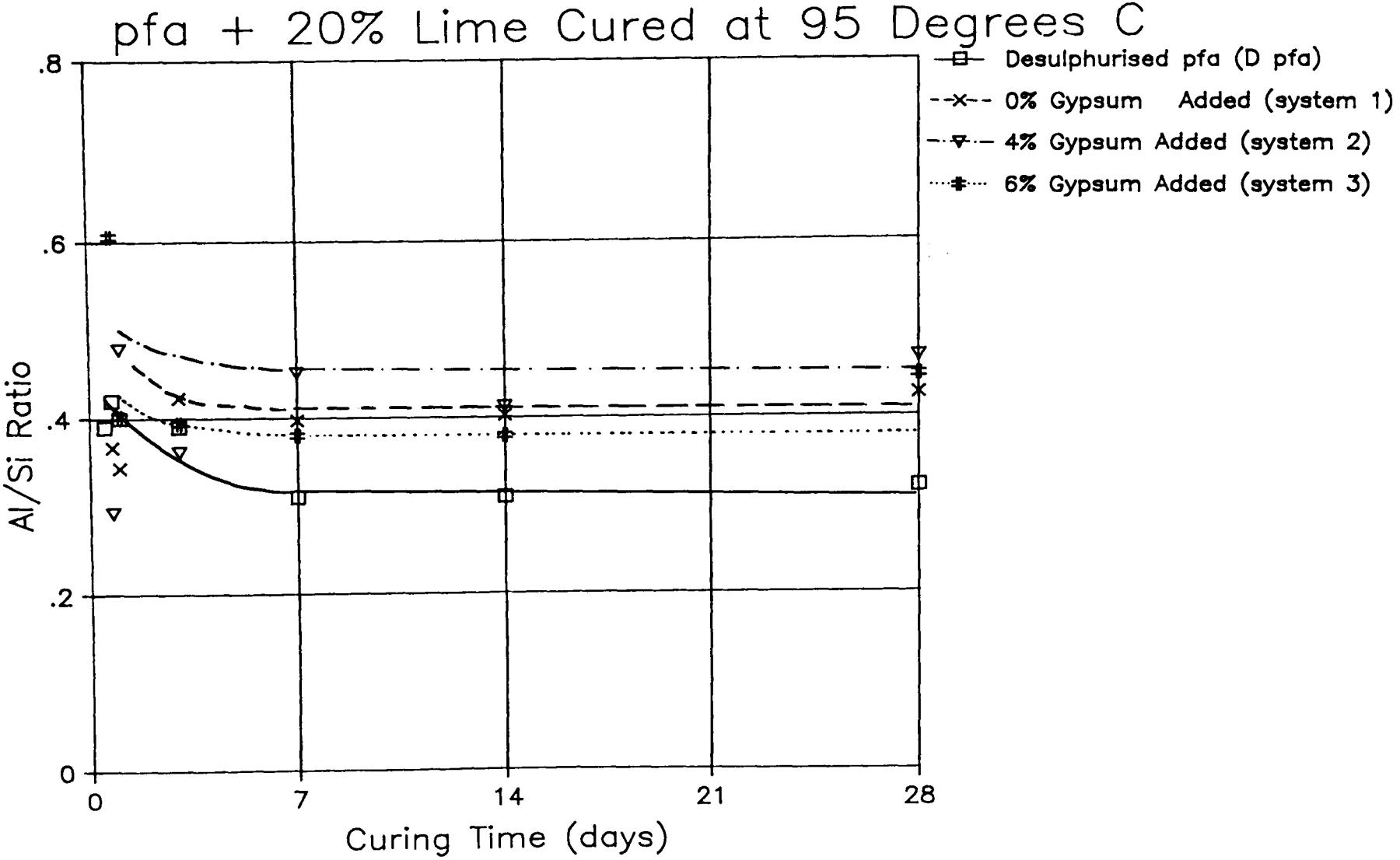


Figure 8.14 Al/Si ratio versus curing time (up to 28 days) for system D pfa, system 1, system 2 and system 3.

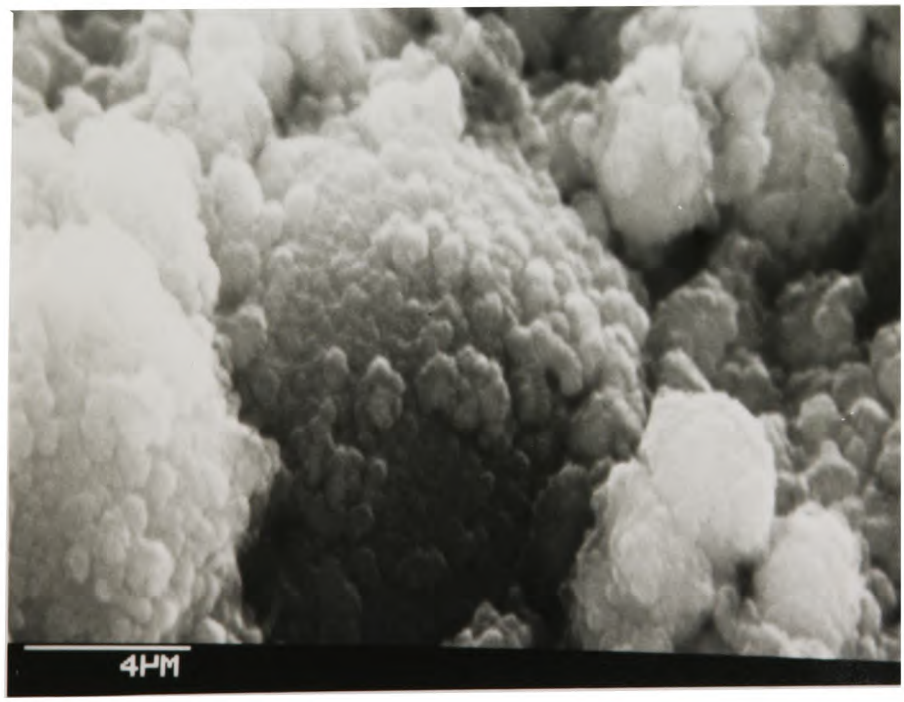


Plate 8.1 SEM micrograph of a system D pfa specimen cured for 3 hours.

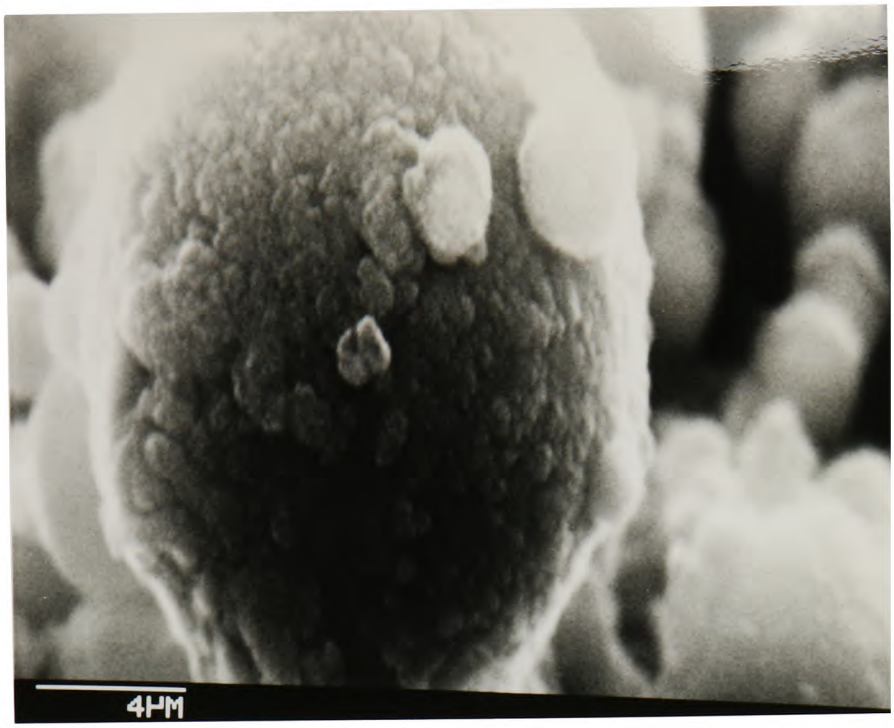


Plate 8.2 SEM micrograph of a system D pfa specimen cured for 6 hours.

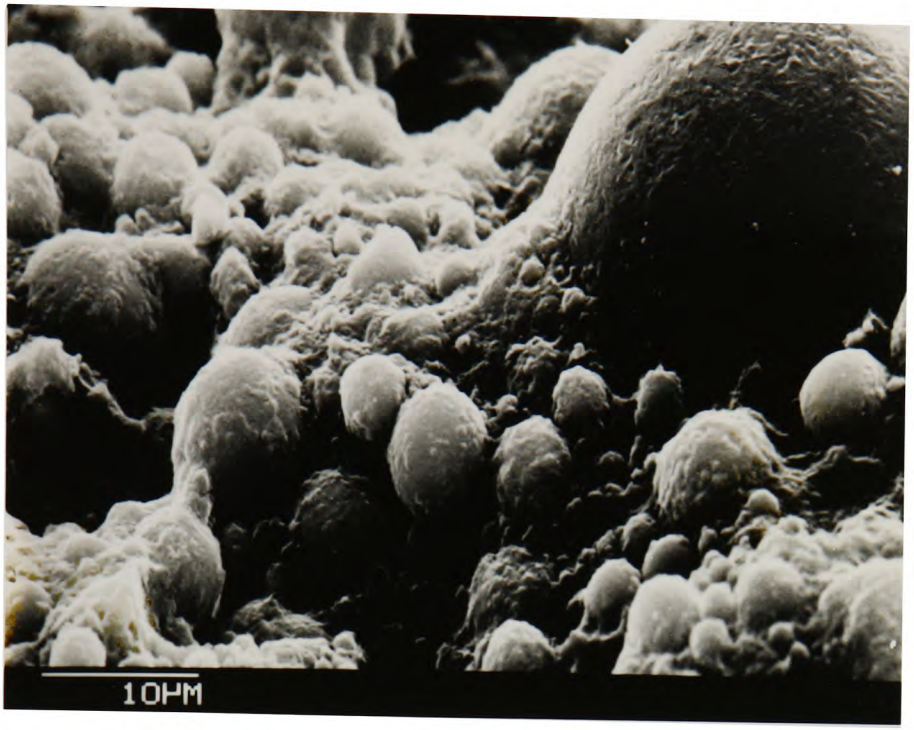


Plate 8.3 SEM micrograph of a system D pfa specimen cured for 12 hours.

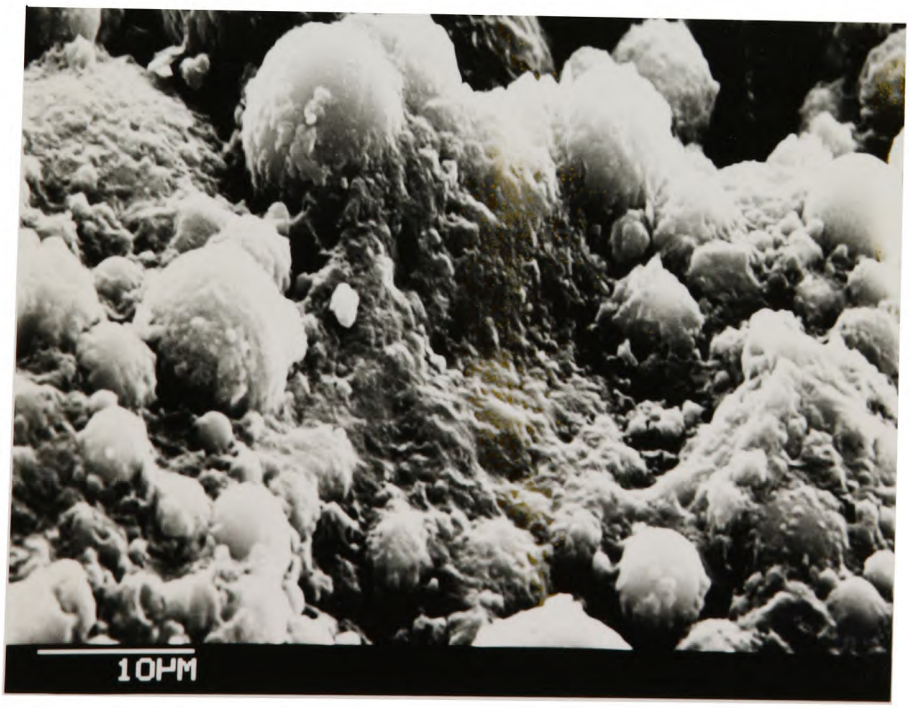


Plate 8.4 SEM micrograph of a system D pfa specimen cured for 18 hours.

Plate 8.5 SEM micrograph of a system D pfa specimen cured for 24 hours.

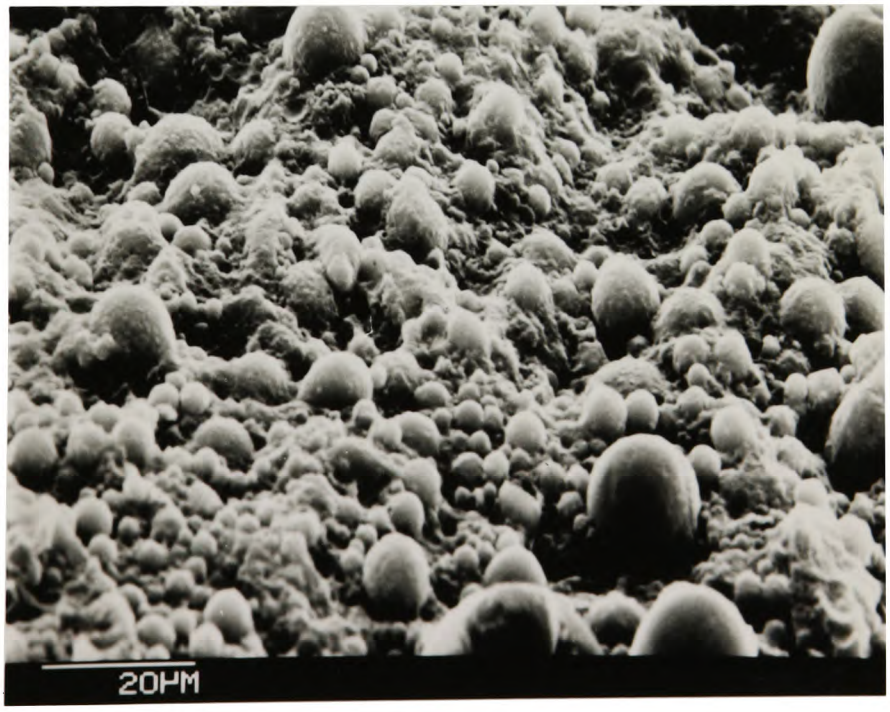
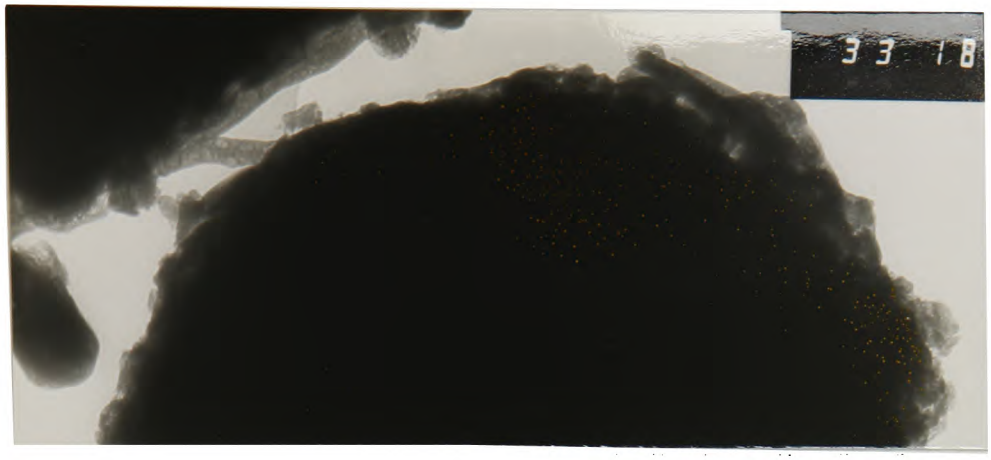


Plate 8.6 SEM micrograph of a system D pfa specimen cured for 7 days.



152 nm

Plate 8.7 TEM micrograph showing surface reaction product - system D pfa, specimen cured for 3 hours.

167 nm

Plate 8.8 TEM micrograph of fibrous gel - system D pfa, specimen cured for 3 hours.

Plate 8.9 TEM micrograph of surface reaction product - system D pfa, specimen cured for 6 hours.

Plate 8.10 TEM micrograph of fibrous gel - system D pfa, specimen cured for 6 hours.

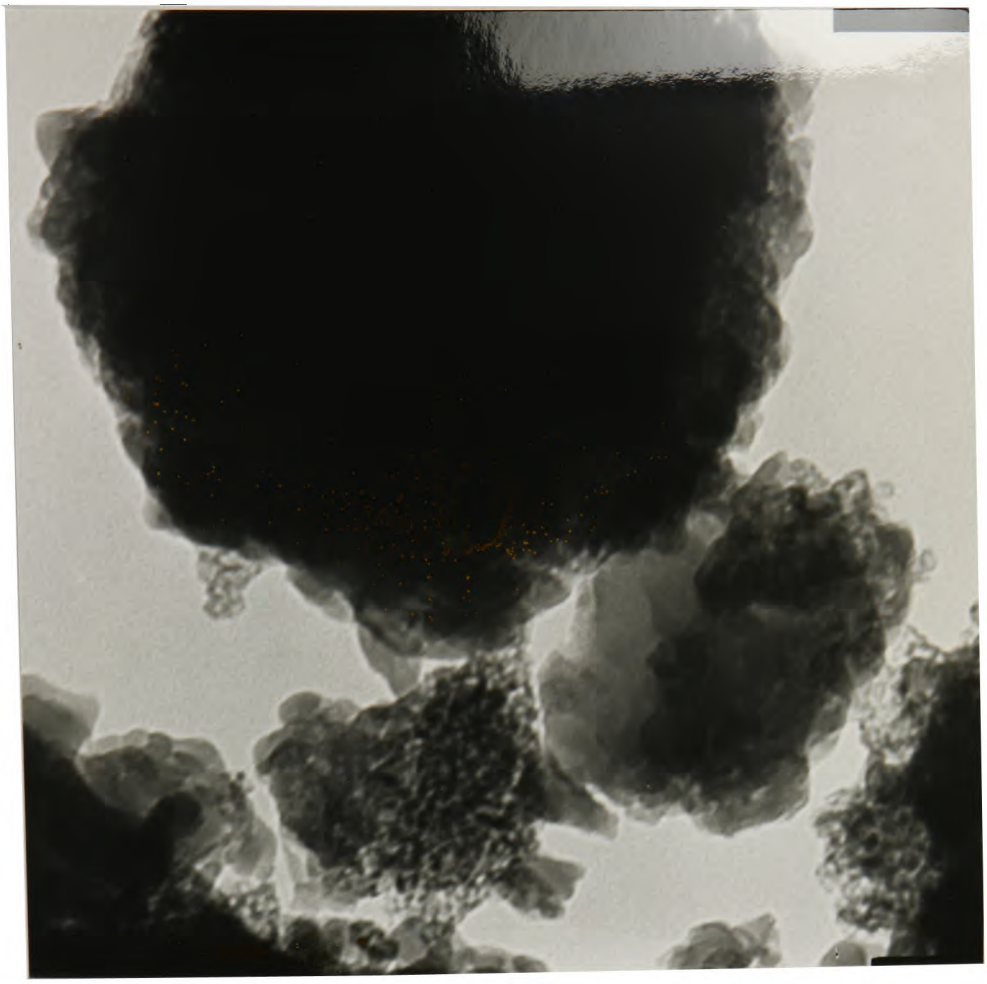


Plate 8.11 TEM micrograph of surface reaction product - system D pfa, specimen cured for 12 hours.

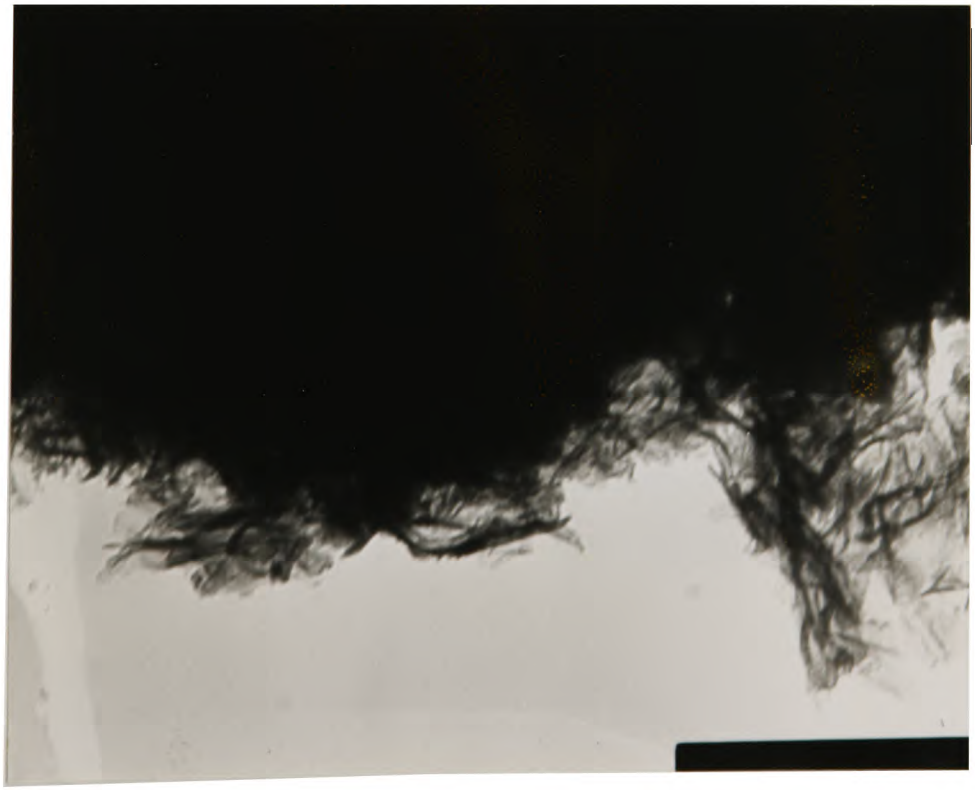


Plate 8.12 TEM micrograph of fibrous gel - system D pfa, specimen cured for 12 hours.

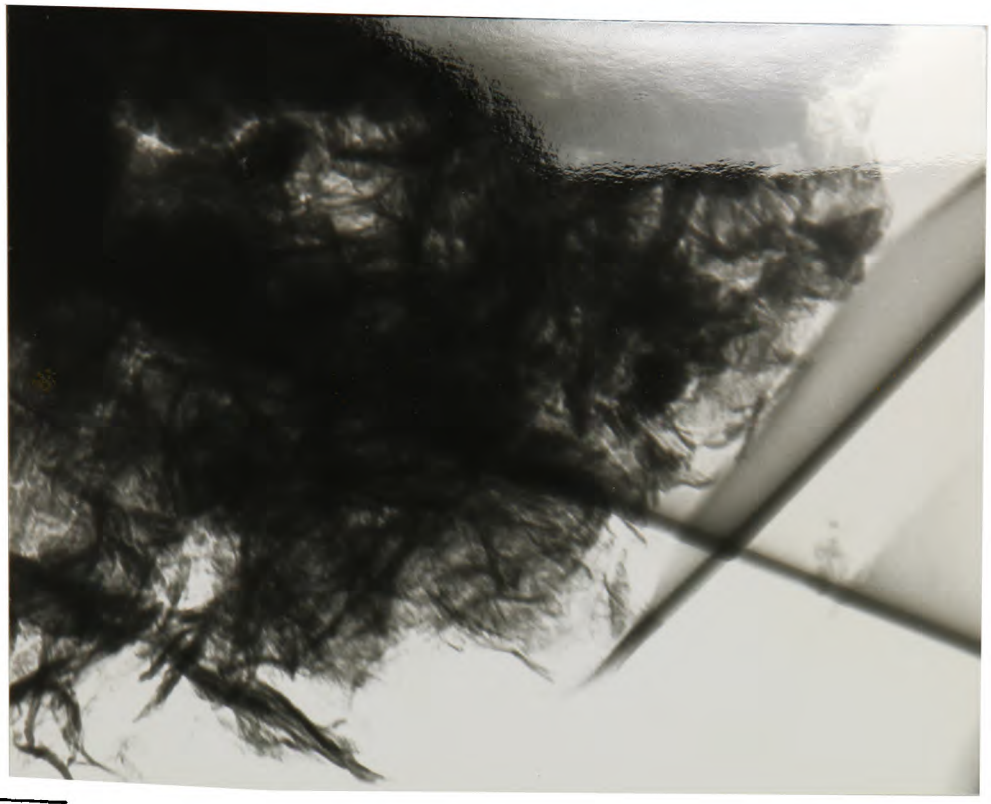
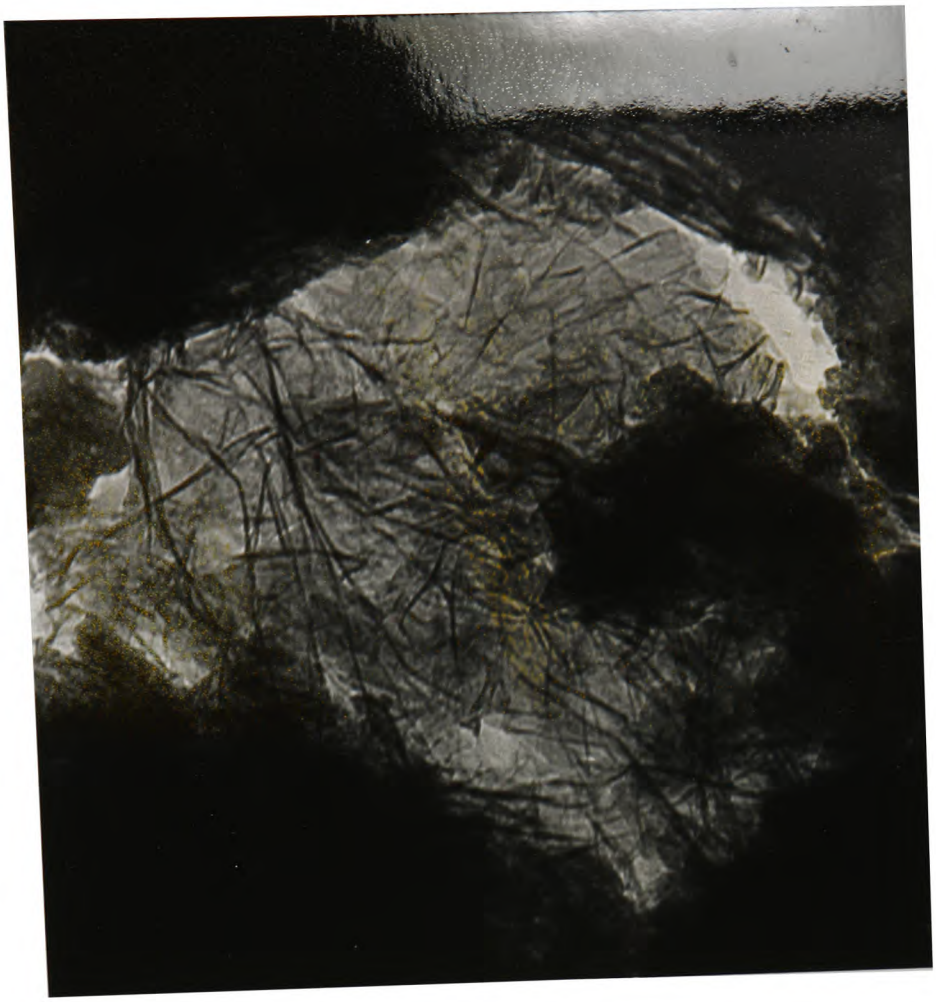


Plate 8.13 TEM micrograph of fibrous gel - system D pfa, specimen cured for 24 hours.

Plate 8.14 TEM micrograph of fibrous gel - system D pfa, specimen cured for 3 days.



67nm

Plate 8.15 TEM micrograph of fibrous gel - system D pfa, specimen cured for 14 days.

225nm

Plate 8.16 TEM micrograph of fibrous gel - system D pfa, specimen cured for 28 days.

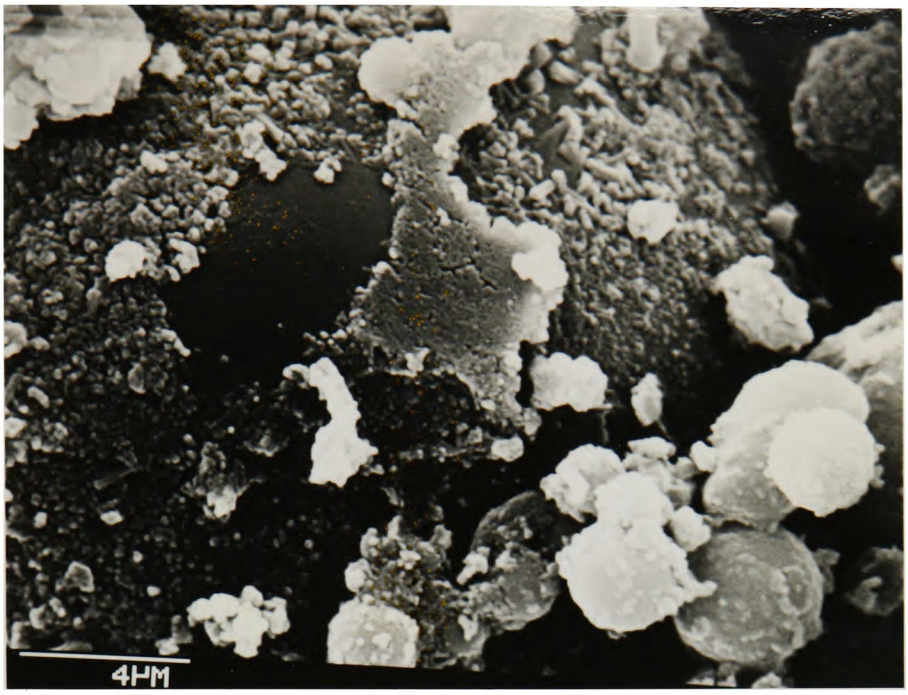


Plate 8.17 SEM micrograph of a system 2 specimen cured for 3 hours.

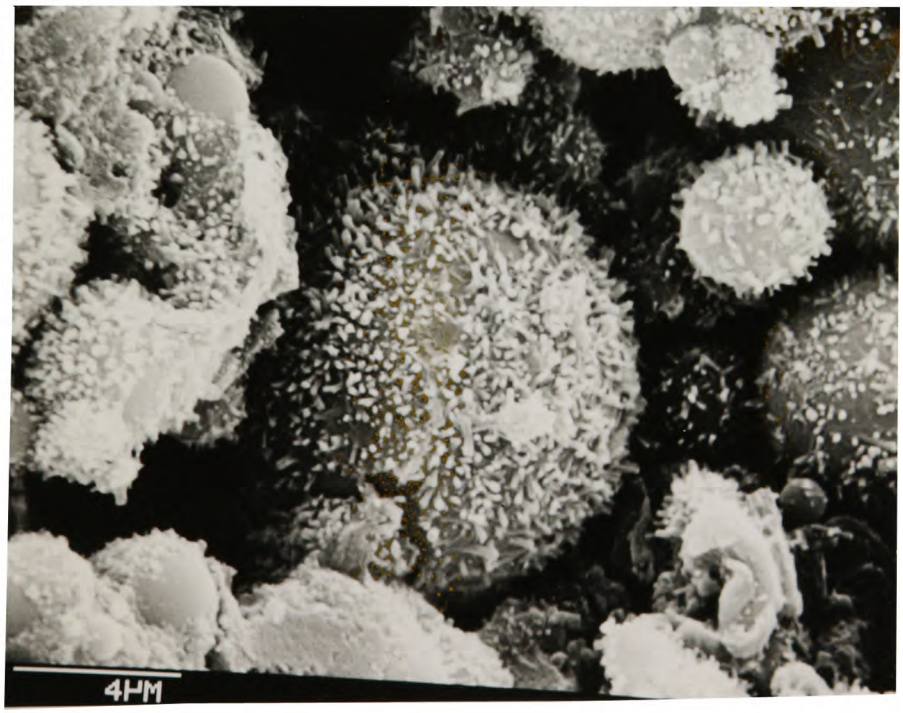


Plate 8.18 SEM micrograph of a system 2 specimen cured for 6 hours.

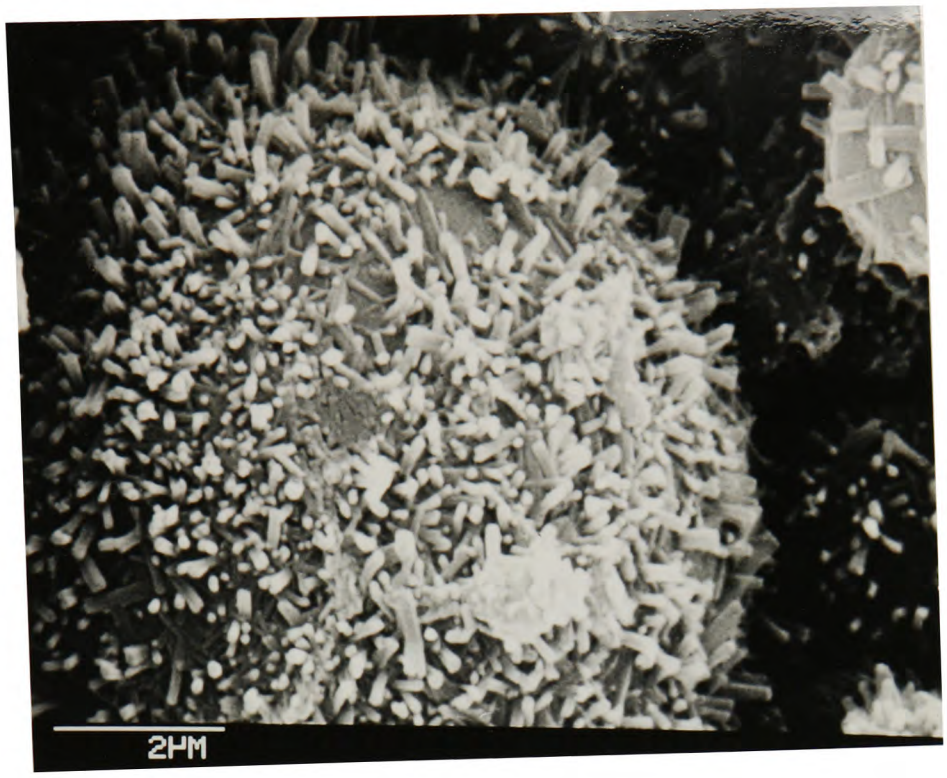


Plate 8.19 SEM micrograph showing ettringite coating system 2 specimen cured for 6 hours.

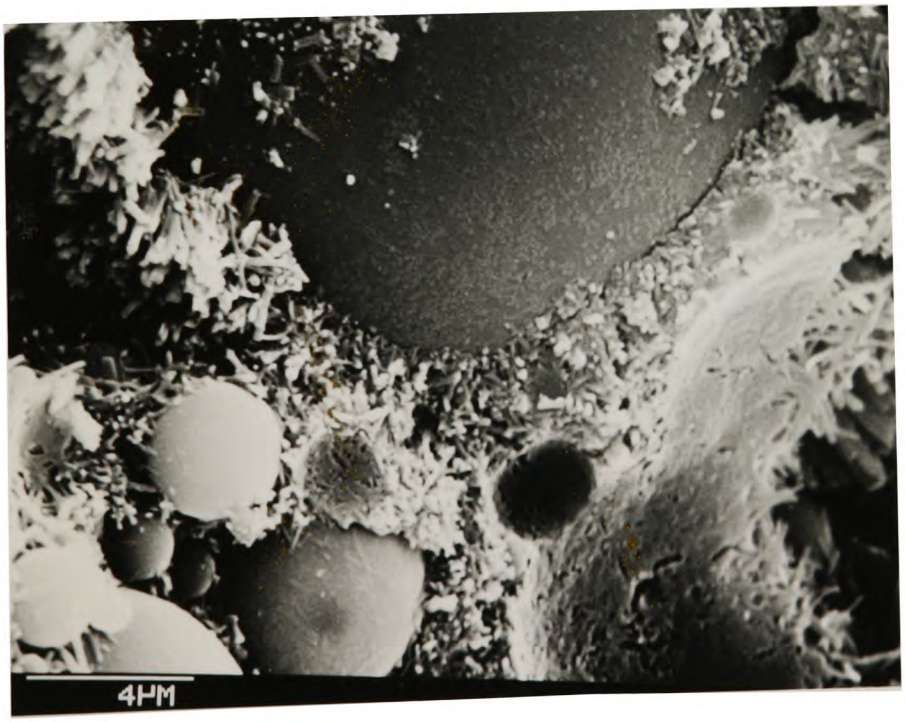


Plate 8.20 SEM micrograph showing ettringite coating in section - system 2 specimen cured for 6 hours.

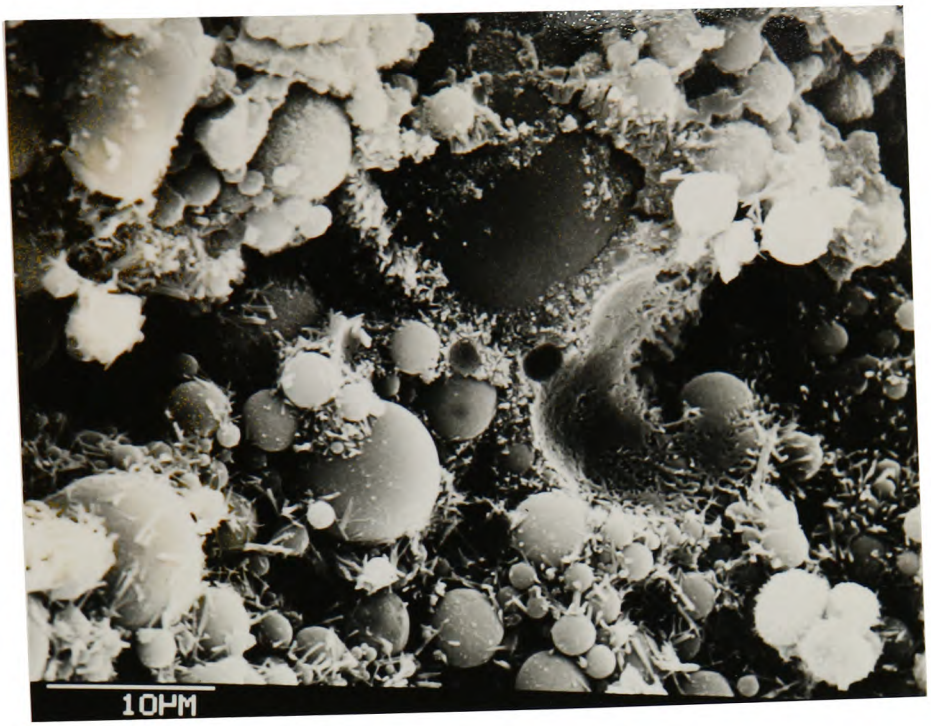


Plate 8.21 SEM micrograph showing network of ettringite rods - system 2 specimen cured for 6 hours.

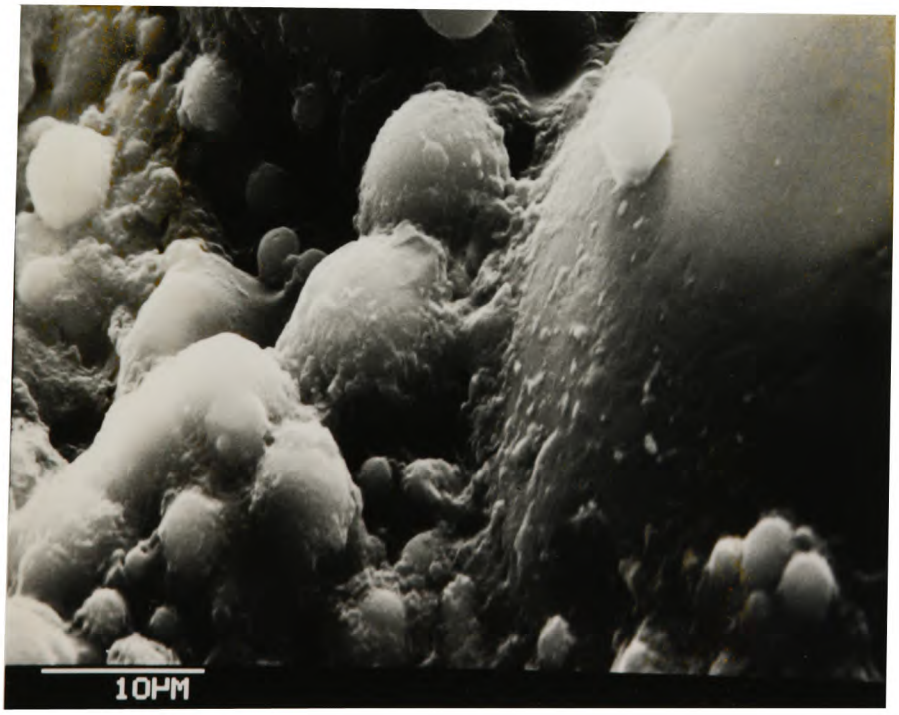


Plate 8.22 SEM micrograph of a system 2 specimen cured for 12 hours.

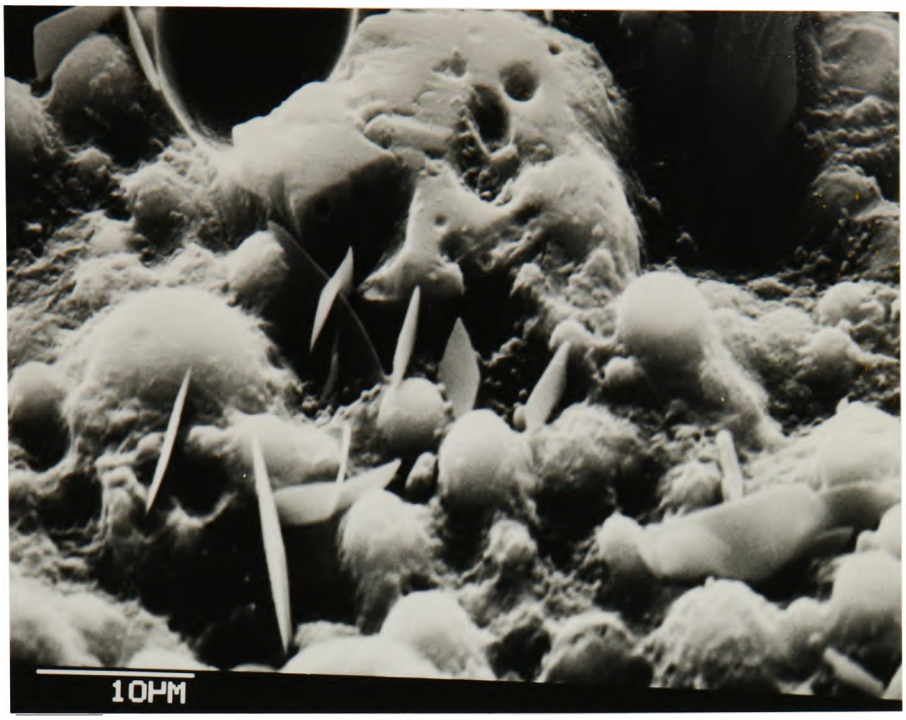


Plate 8.23 SEM micrograph of a system 2 specimen cured for 24 hours.

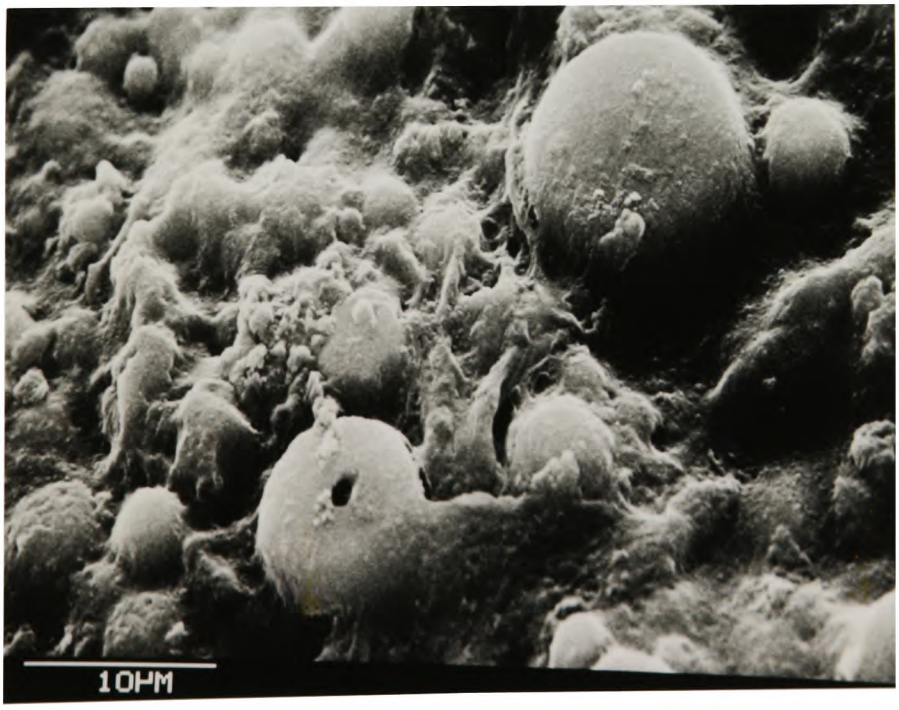


Plate 8.24 SEM micrograph of a system 2 specimen cured for 3 days.

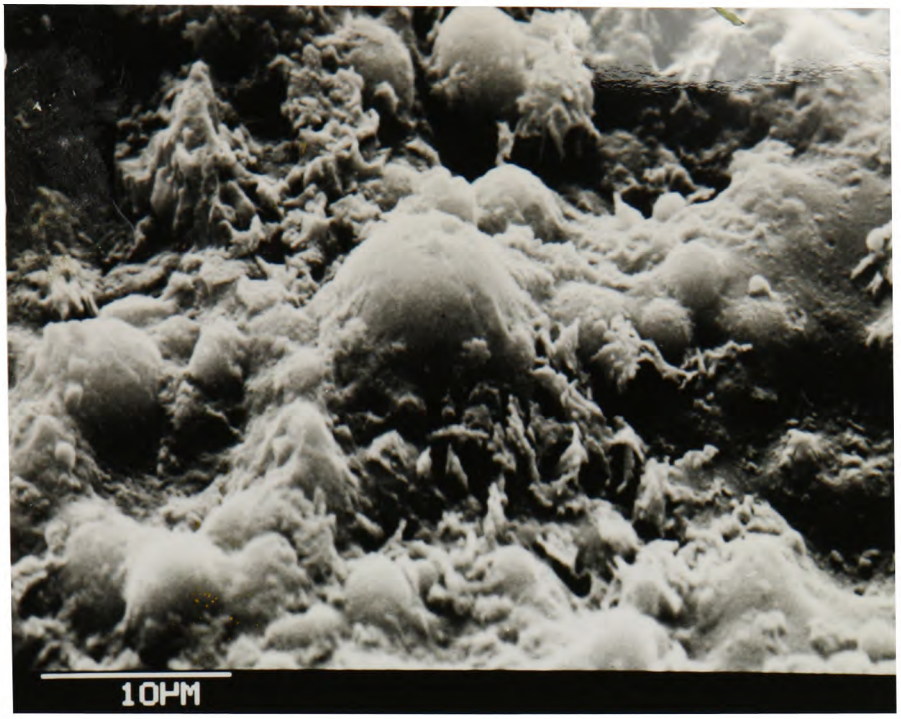


Plate 8.25 SEM micrograph of a system 2 specimen cured for 7 days.

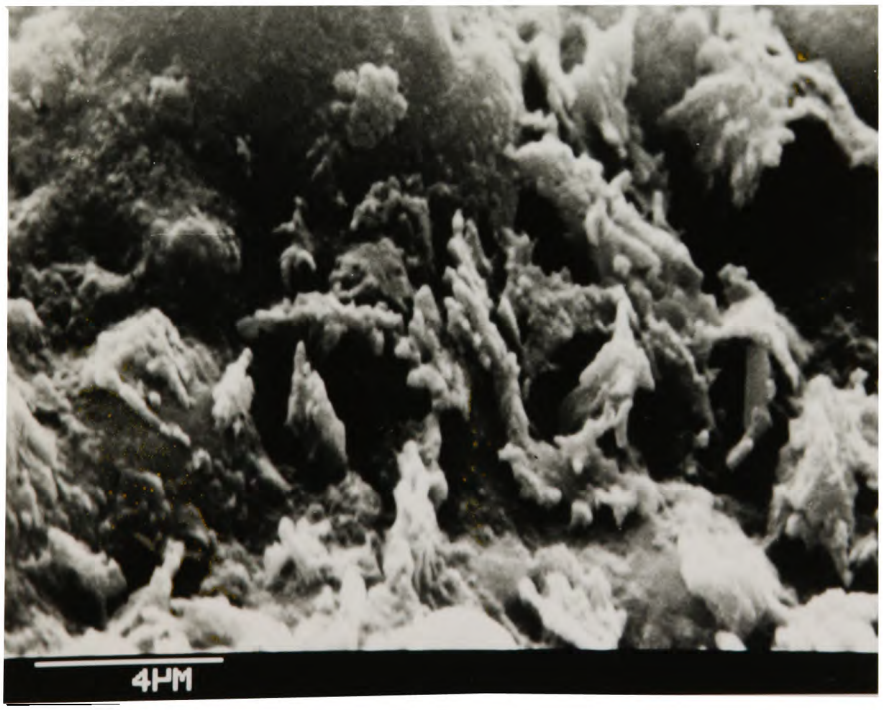


Plate 8.26 SEM micrograph of a system 2 specimen cured for 7 days.

Plate 8.27 SEM micrograph of a system 2 specimen cured for 14 days.

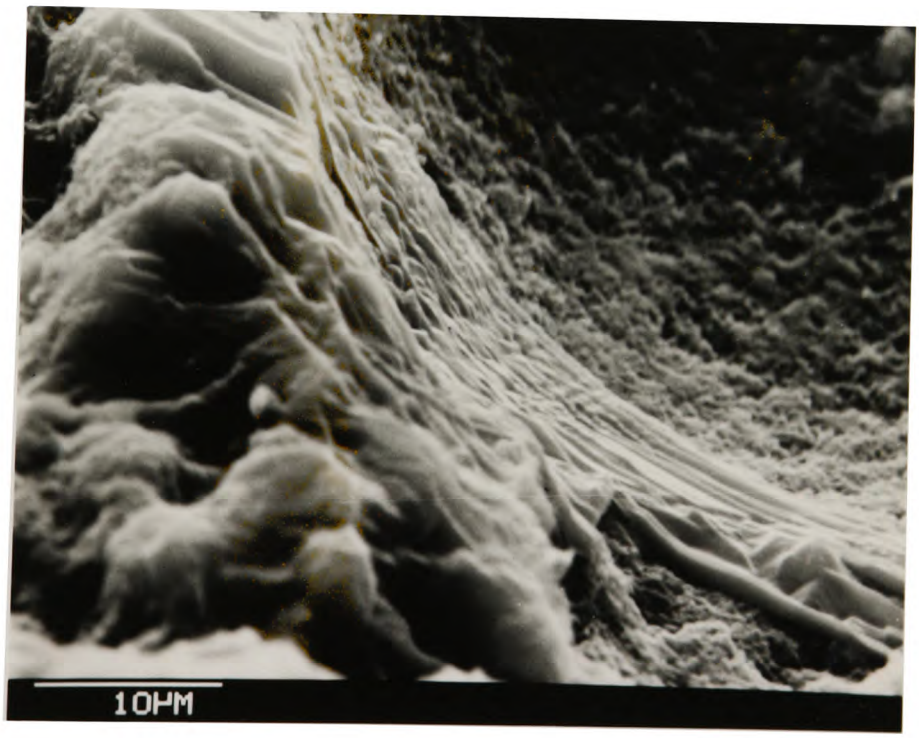


Plate 8.28 SEM micrograph of a system 2 specimen cured for 28 days.

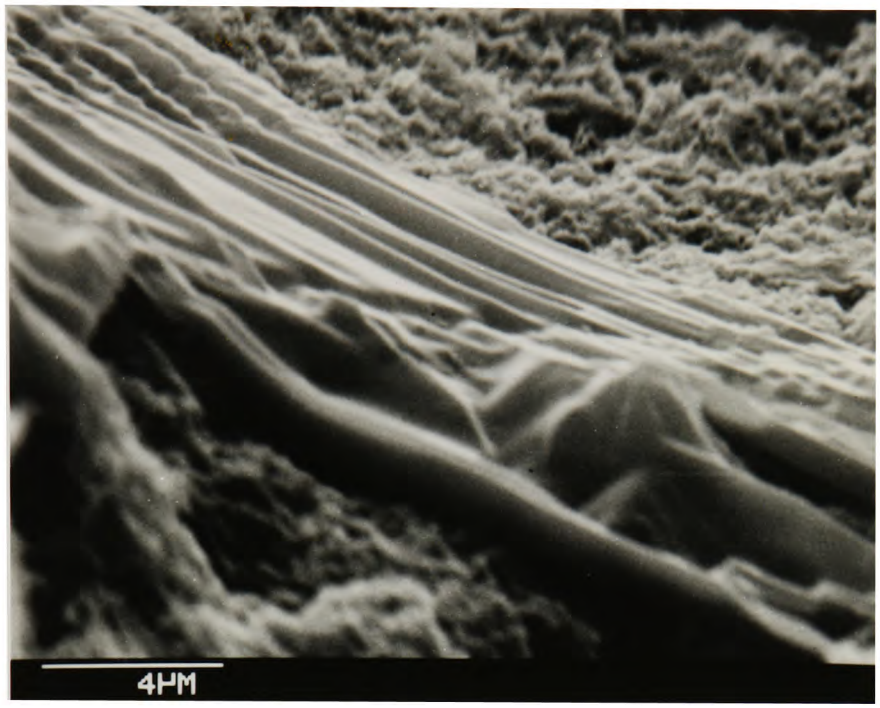
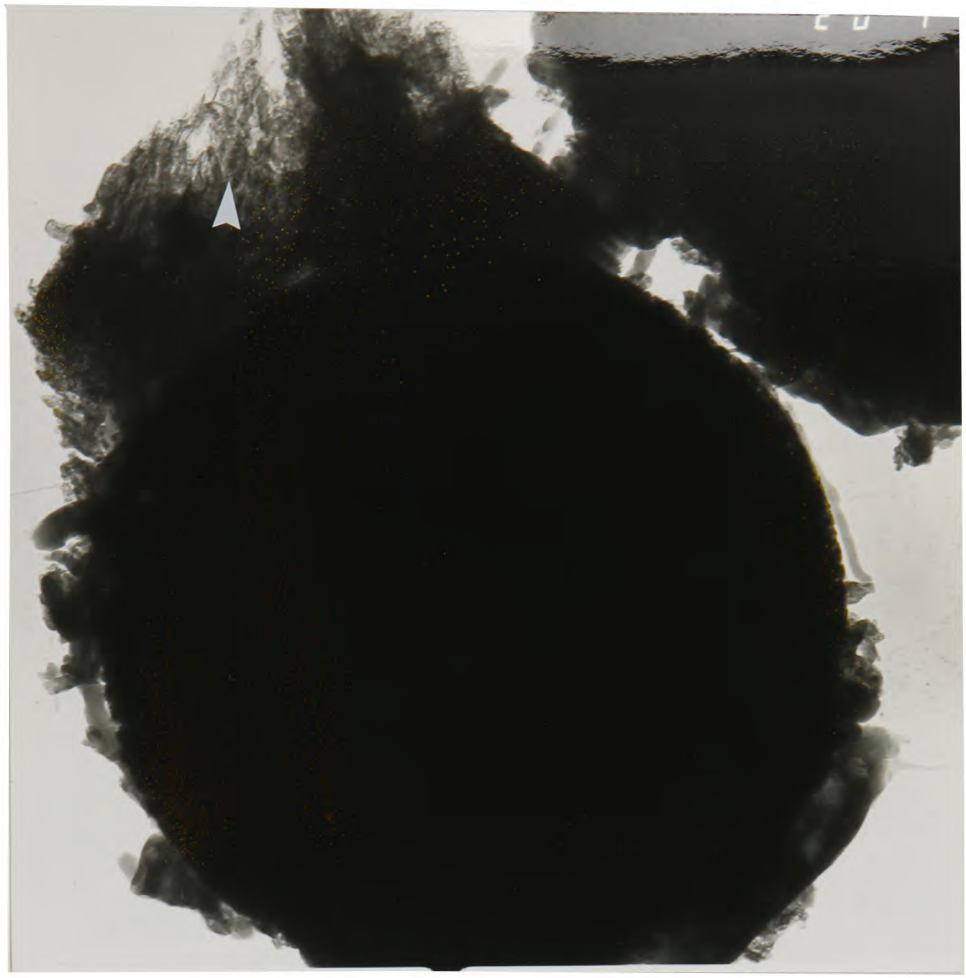


Plate 8.29 SEM micrograph of a system 2 specimen cured for 28 days.

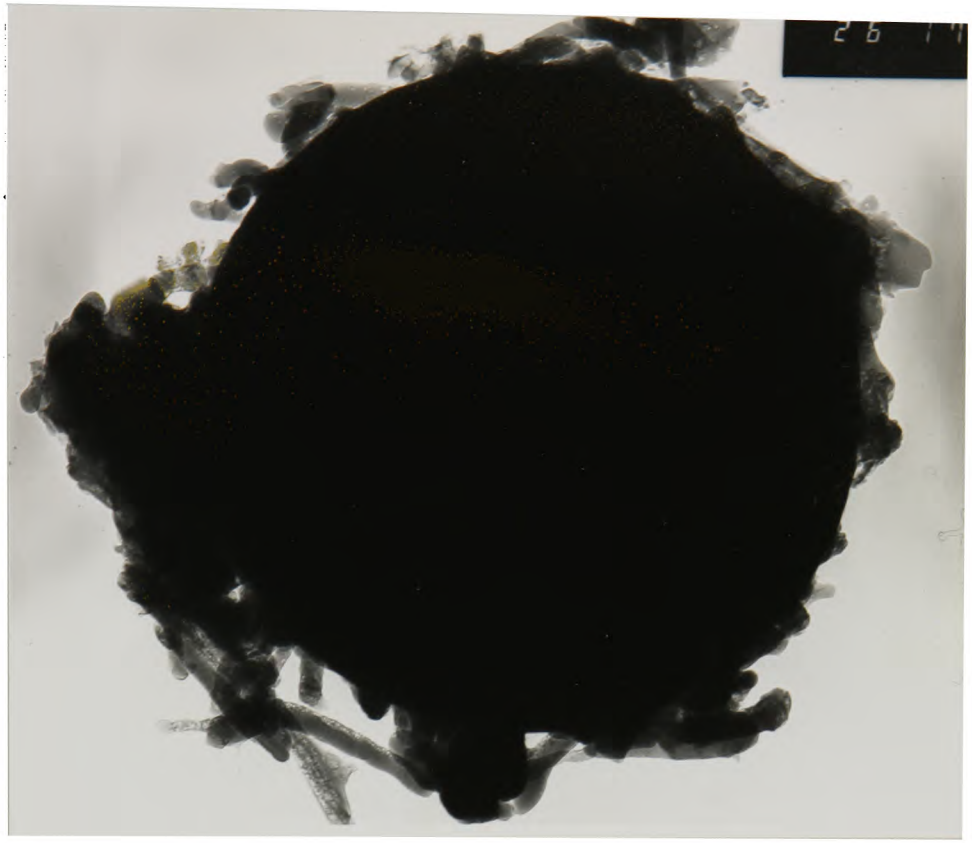


240nm

EDAX Results

E	%	Ratio to Si
Na	2.5	0.29
Al	1.52	0.16
Si	9.50	-
S	1.19	0.12
K	0.54	0.05
Ca	82.60	8.70
Fe	1.81	0.19

Plate 8.30 TEM micrograph showing surface reaction product - system 2 specimen cured for 12 hours.



205nm

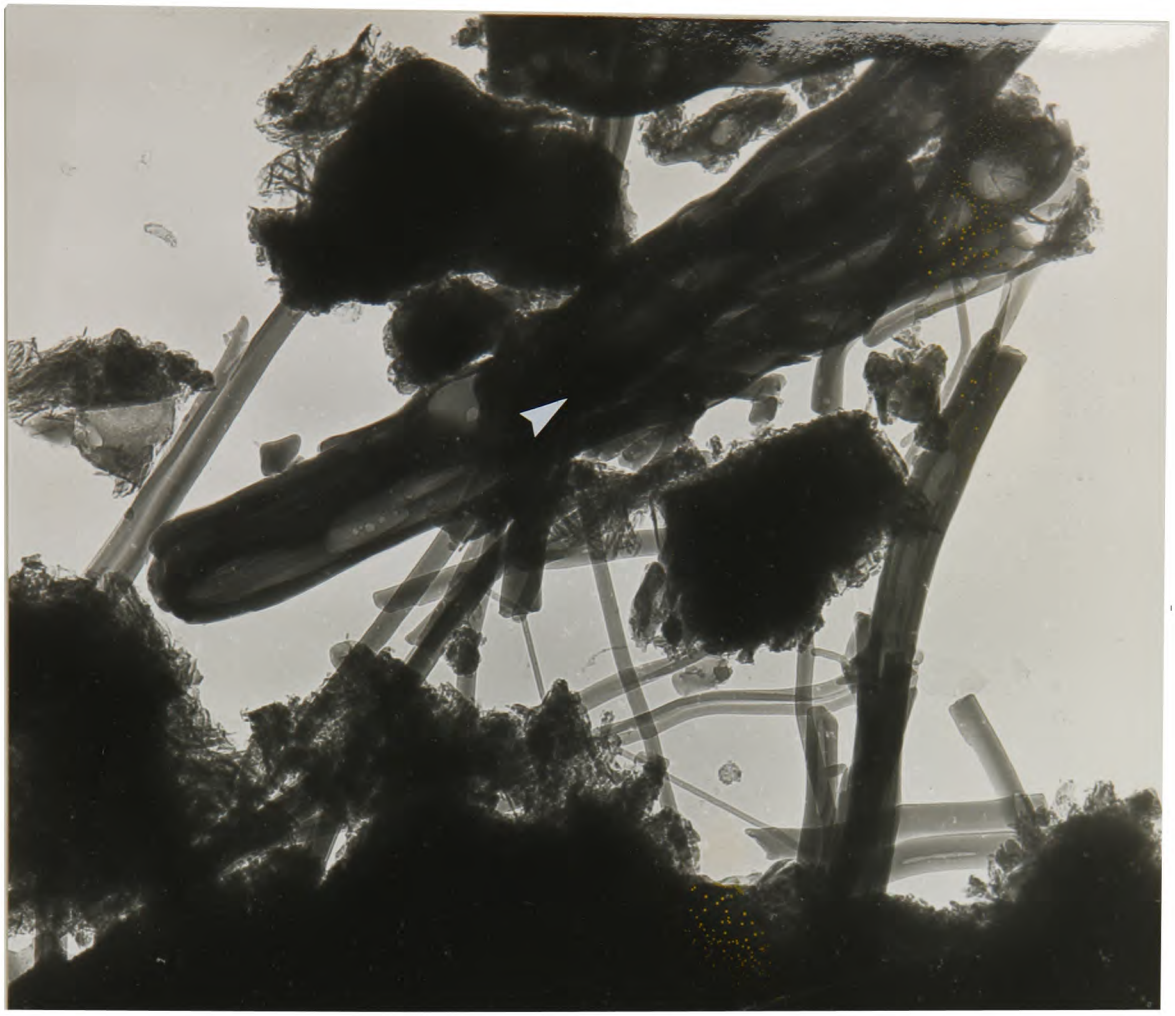
Plate 8.31 TEM micrograph showing surface ettringite rods - system 2 specimen cured for 12 hours.

310 nm

EDAX Results

E	%	Theoretical ettringite composition (%)
Na	-	-
Al	29.1	18.2
Si	1.31	-
S	24.80	27.3
K	0.56	-
Ca	43.7	54.5
Fe	0.33	-

Plate 8.32 TEM micrograph showing ettringite rods - system 2 specimen cured for 18 hours.

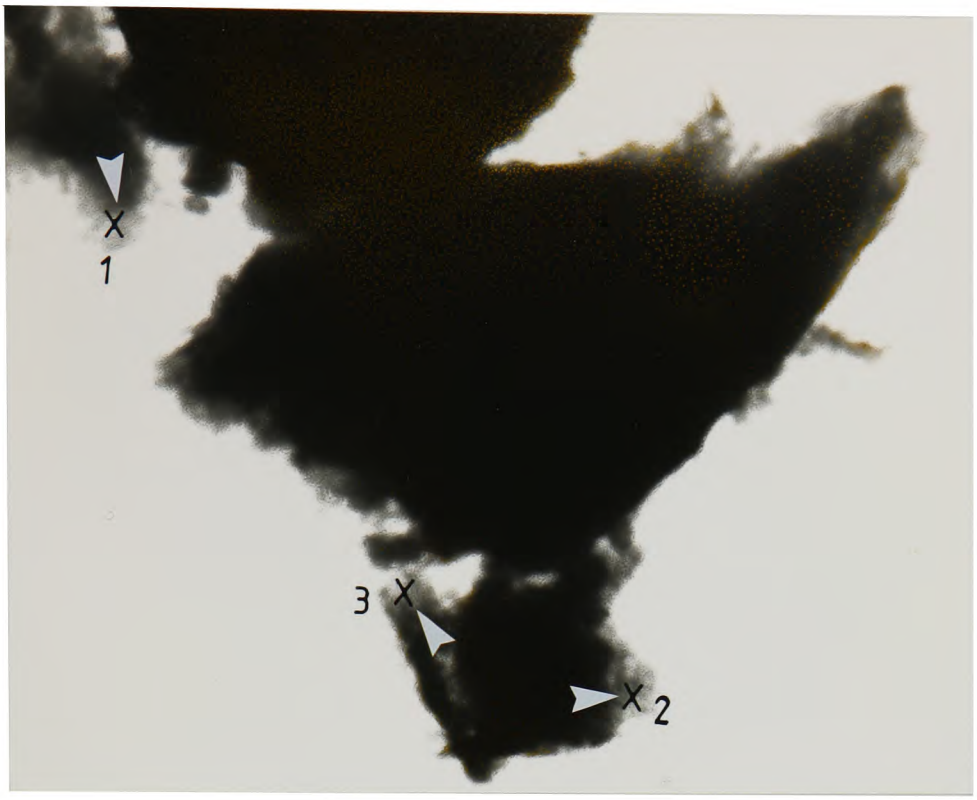


256nm

EDAX Results

E	%	Theoretical ettringite composition (%)
Na	-	-
Al	29.0	18.2
Si	2.54	-
S	21.32	27.3
K	0.68	-
Ca	42.7	54.5
Fe	0.58	-

Plate 8.33 TEM micrograph showing tubular growth - system 2 specimen cured for 18 hours.

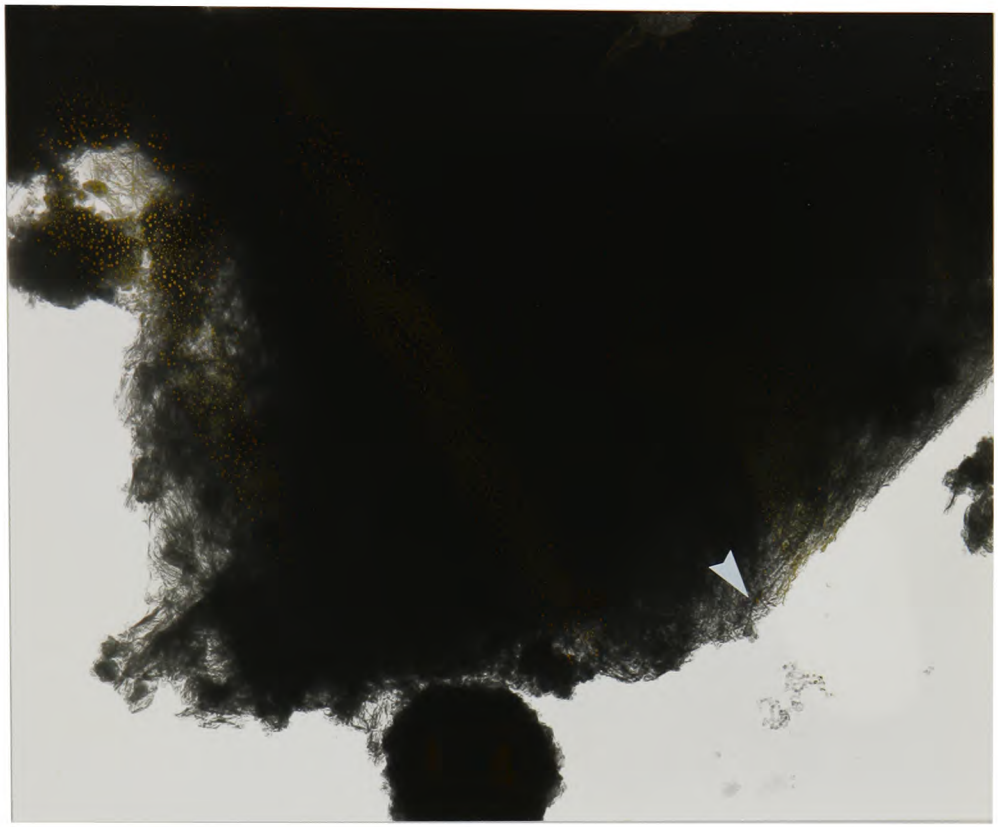


208 nm

EDAX Results

E	Analysis 1 %	Analysis 1 Ratio to Si	Analysis 2 %	Analysis 2 Ratio to Si	Analysis 3 %	Analysis 3 Ratio to Si
Na	5.27	0.12	2.12	0.08	2.57	0.05
Al	11.3	0.27	23.9	0.93	8.80	0.19
Si	41.5	-	25.6	-	45.5	-
S	2.91	0.07	8.17	0.31	3.19	0.07
K	2.74	0.06	1.29	0.05	1.53	0.03
Ca	33.9	0.81	36.6	1.42	37.2	0.81
Fe	2.27	0.05	2.15	0.08	1.03	0.02

Plate 8.34 TEM micrograph showing colloidal reaction product - system 2 specimen cured for 18 hours.

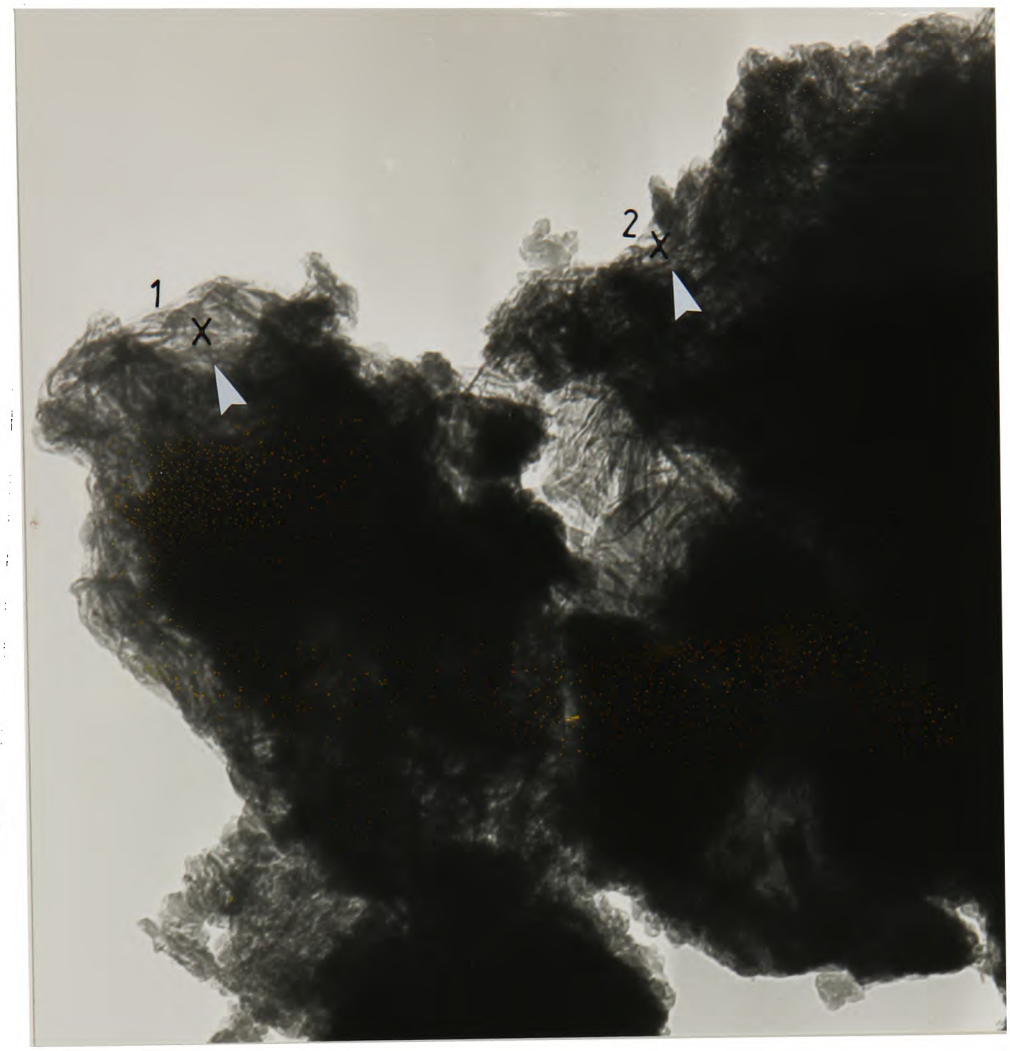


417nm

EDAX Results

E	%	Ratio to Si
Na	3.36	0.08
Al	11.4	0.28
Si	39.9	-
S	3.97	0.10
K	4.56	0.11
Ca	35.17	0.88
Fe	1.50	0.03

Plate 8.35 TEM micrograph showing colloidal and fibrous reaction product - system 2 specimen cured for 18 hours.

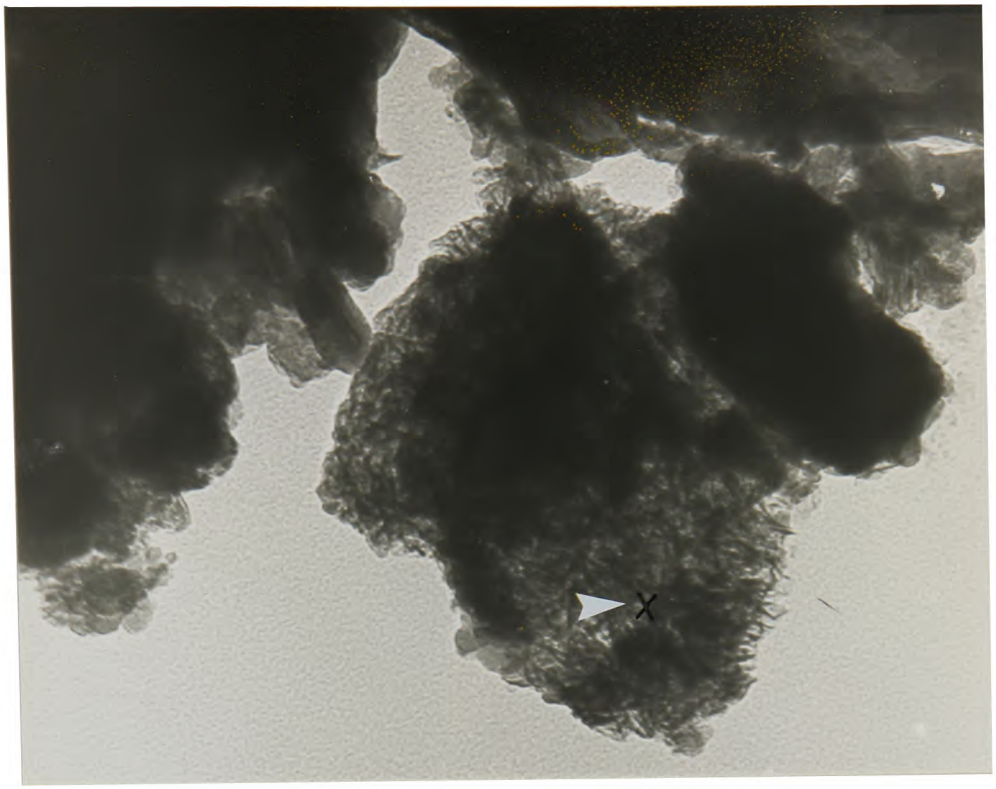


107 nm

EDAX Results

E	Analysis 1 %	Analysis 1 Ratio to Si	Analysis 2 %	Analysis 2 Ratio to Si
Na	-	-	2.12	0.07
Al	15.33	0.425	22.0	0.77
Si	36.0	-	28.5	-
S	4.82	0.13	6.17	0.21
K	1.72	0.04	3.97	0.14
Ca	34.0	0.94	35.7	1.25
Fe	1.23	0.03	1.42	0.05

Plate 8.36 TEM micrograph showing fibrous gel - system 2 specimen cured for 24 hours.

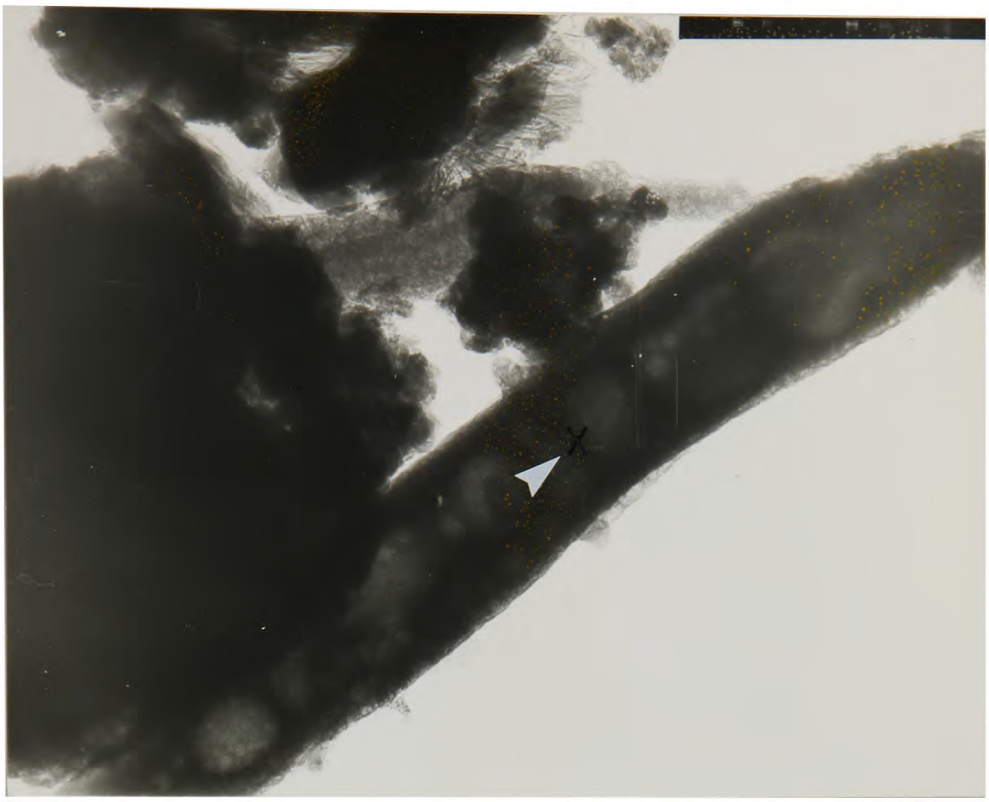


97nm

EDAX Results

E	%	Ratio to Si
Na	2.15	0.05
Al	16.45	0.38
Si	42.5	-
S	2.34	0.05
K	1.19	0.02
Ca	33.60	0.79
Fe	1.69	0.04

Plate 8.37 TEM micrograph of fibrous gel - system 2 specimen cured for 24 hours.

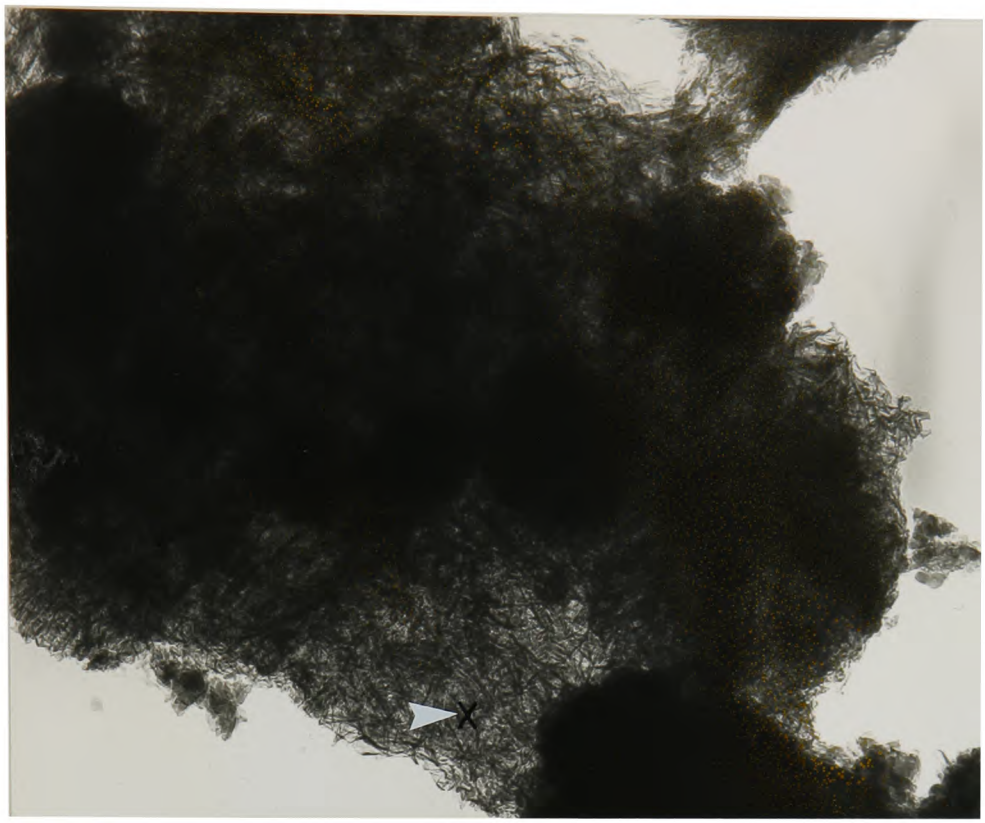


224 nm

EDAX Results

E	%	Theoretical ettringite composition (%)
Na	-	-
Al	29.80	18.2
Si	0.55	-
S	26.1	27.3
K	0.47	-
Ca	42.60	54.5
Fe	0.37	-

Plate 8.38 TEM micrograph showing ettringite rod - system 2 specimen cured for 24 hours.



202 nm

EDAX Results

E	%	Ratio to Si
Na	-	-
Al	19.06	0.43
Si	43.6	-
S	1.87	0.04
K	1.28	0.03
Ca	29.96	0.68
Fe	4.18	0.09

Plate 8.39 TEM micrograph of showing fibrous gel - system 2 specimen cured for 3 days.

105 nm

EDAX Results

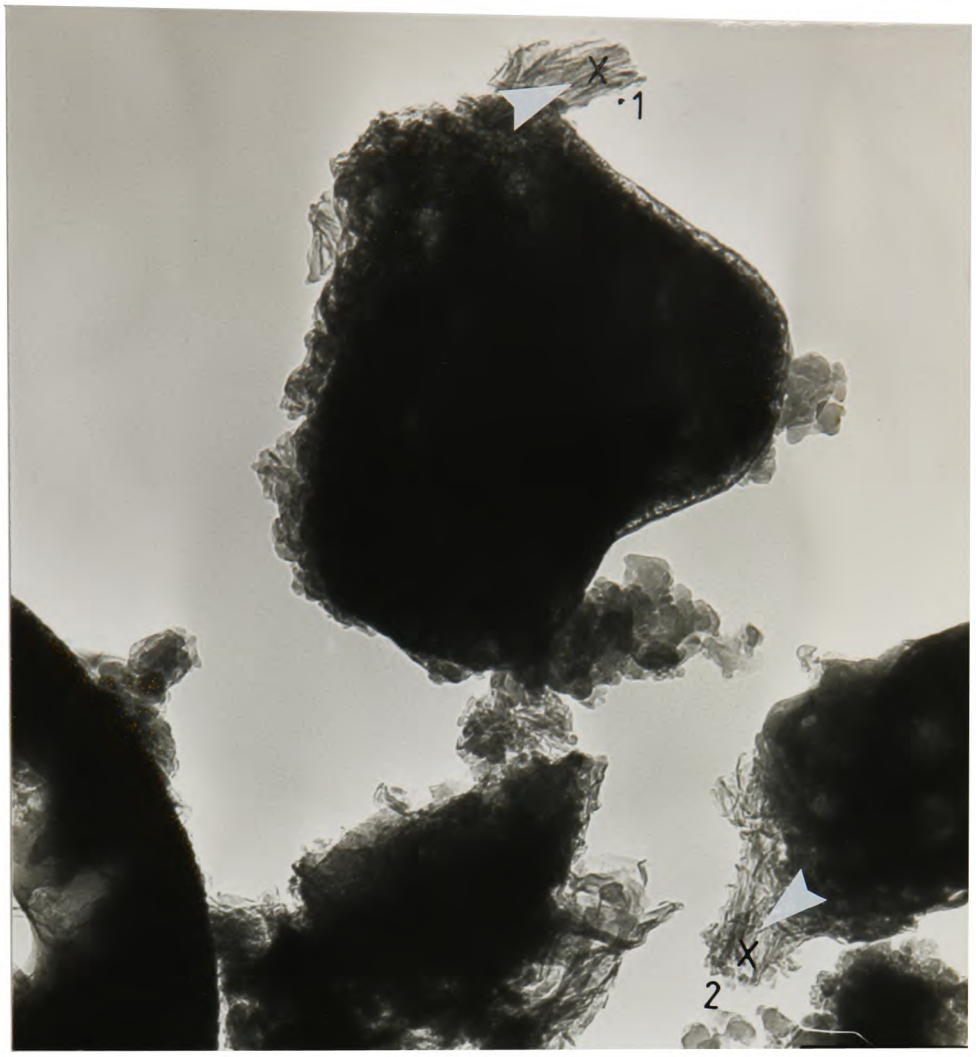
E	Analysis 1 %	Analysis 1 Ratio to Si	Analysis 2 %	Analysis 2 Ratio to Si
Na	1.21	0.04	-	-
Al	11.0	0.36	14.3	0.45
Si	30.7	-	31.40	-
S	3.38	0.11	3.75	0.12
K	3.95	0.12	3.66	0.11
Ca	43.74	1.42	30.60	0.97
Fe	2.73	0.08	2.88	0.09

Plate 8.40 TEM micrograph of showing fibrous gel - system 2 specimen cured for 7 days.

EDAX Results

E	Analysis 1 %	Analysis 1 Ratio to Si	Analysis 2 %	Analysis 2 Ratio to Si
Na	1.06	0.03	2.09	0.05
Al	14.8	0.43	16.42	0.57
Si	33.65	-	38.26	-
S	4.45	0.09	3.51	0.13
K	4.22	0.11	3.29	0.08
Ca	33.30	0.87	29.68	0.81
Fe	2.11	0.05	2.36	0.06

Plate 8.41 TEM micrograph of reaction product - system 2 specimen cured for 7 days.



177 nm

EDAX Results

E	Analysis 1 %	Analysis 1 Ratio to Si	Analysis 2 %	Analysis 2 Ratio to Si
Na	11.74	0.32	3.47	0.09
Al	17.80	0.49	28.22	0.72
Si	36.38	-	38.81	-
S	2.55	0.07	2.06	0.05
K	2.84	0.07	1.09	0.03
Ca	27.2	0.75	23.30	0.60
Fe	1.40	0.03	2.98	0.07

Plate 8.42 TEM micrograph showing residual reacted shell - system 2 specimen cured for 7 days.

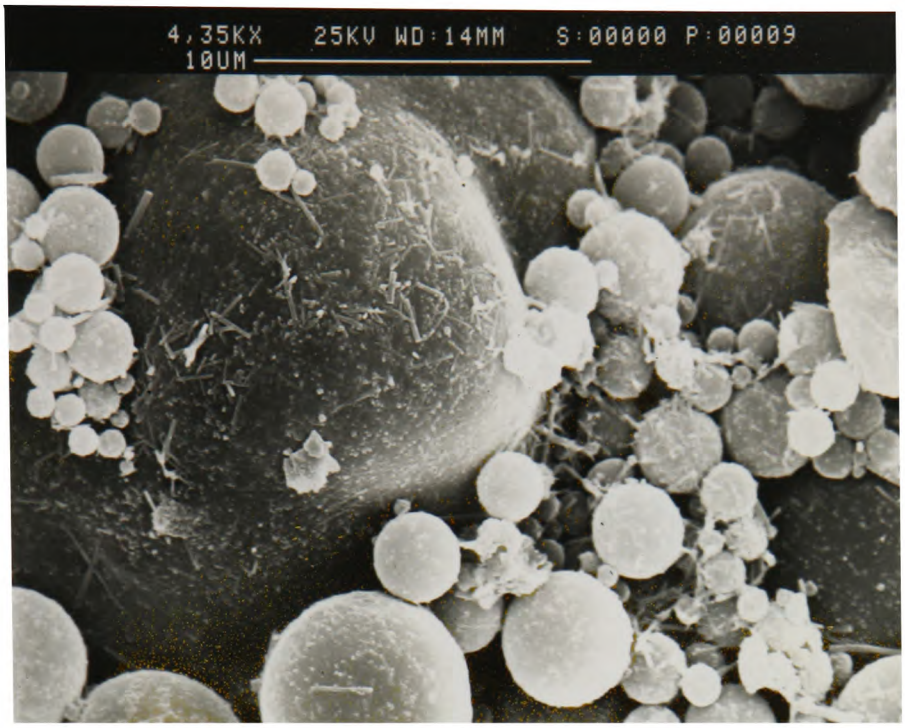


Plate 8.43 SEM micrograph of a system 3 specimen cured for 3 hours.

Plate 8.44 SEM micrograph of a system 3 specimen cured for 6 hours.

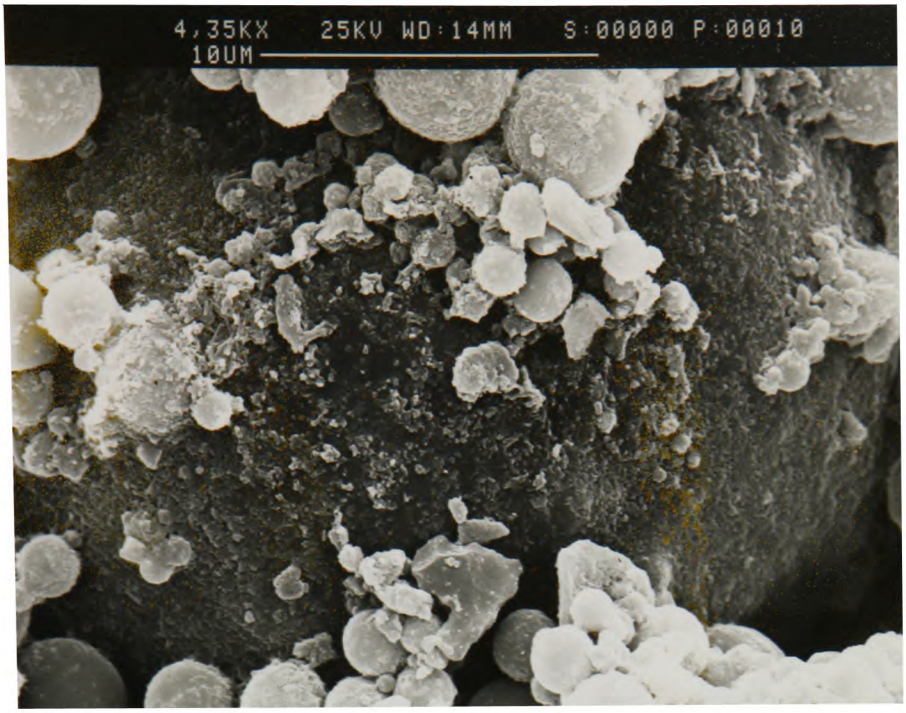


Plate 8.45 SEM micrograph of a system 3 specimen cured for 12 hours.

Plate 8.46 SEM micrograph showing toothed edged gypsum crystals - system 3 specimen cured for 12 hours.

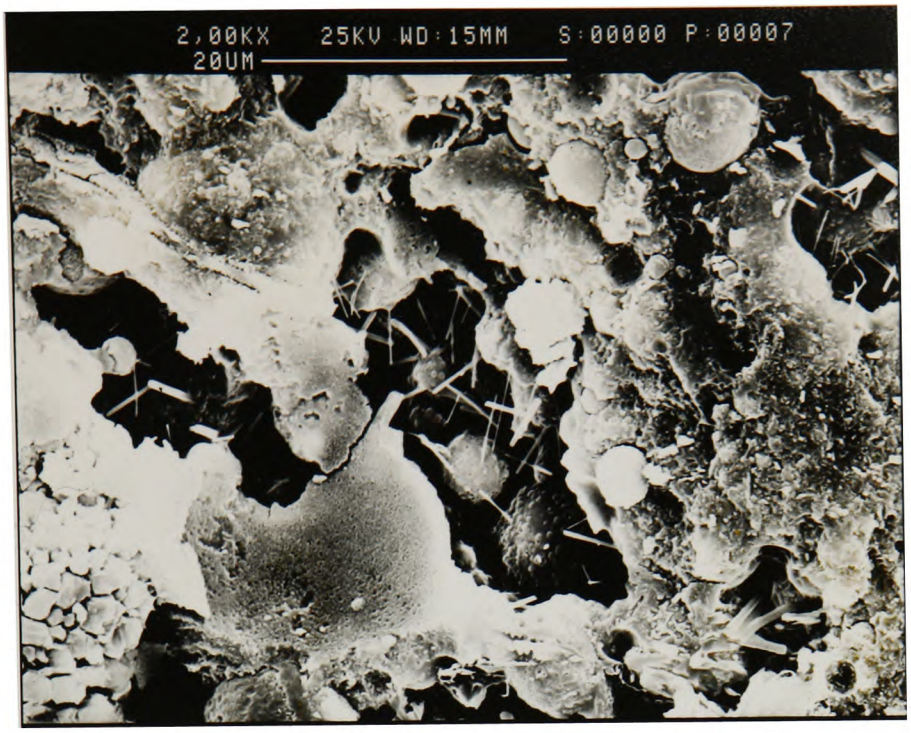


Plate 8.47 SEM micrograph of a system 3 specimen cured for 18 hours.

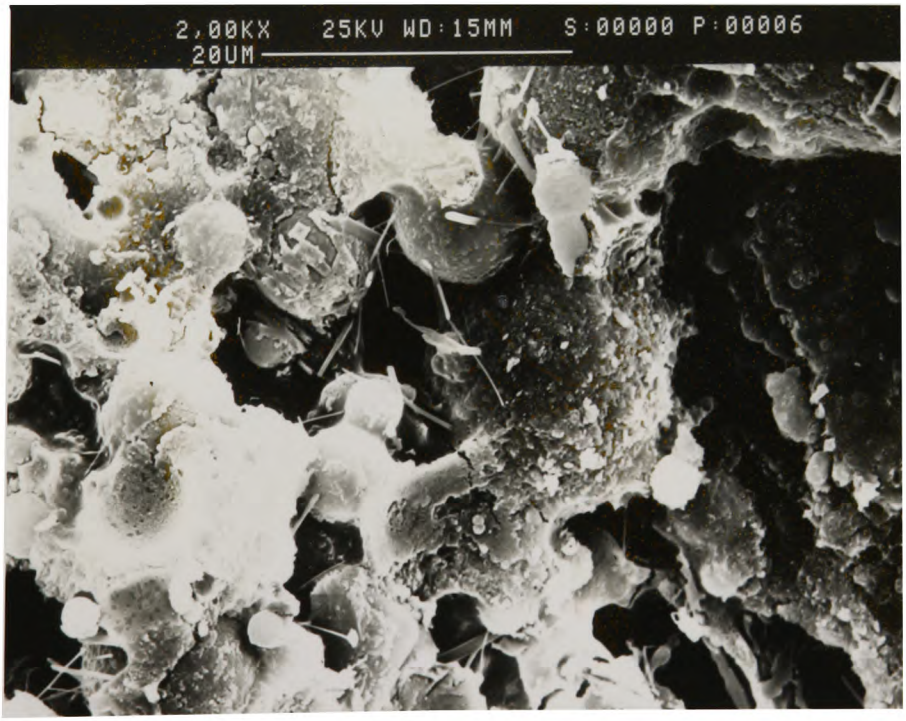


Plate 8.48 SEM micrograph showing typical microstructure - system 3 specimen cured for 18 hours.

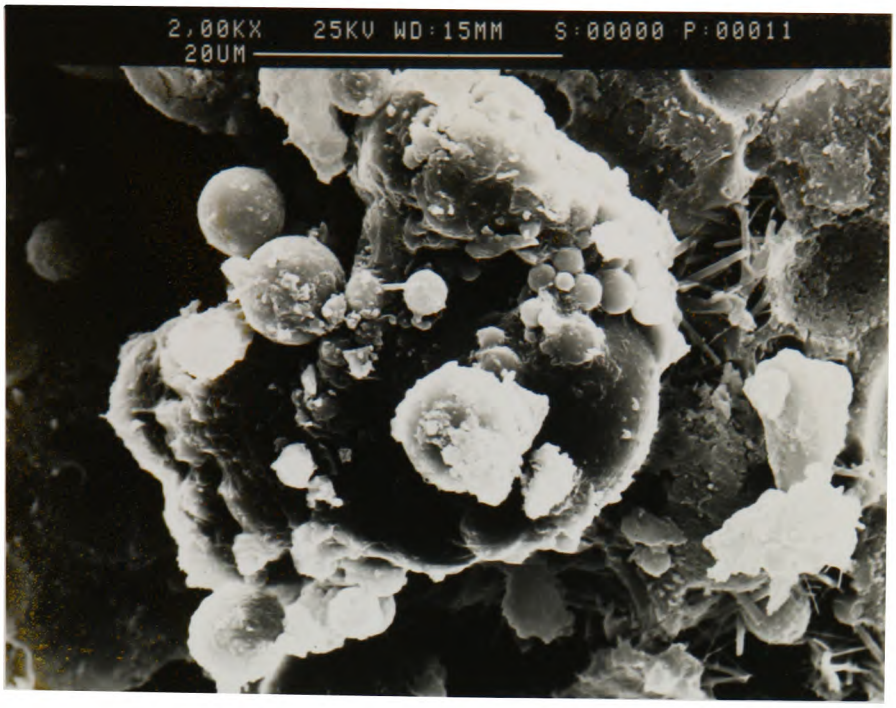


Plate 8.49 SEM micrograph of a system 3 specimen cured for 24 hours.

Plate 8.50 SEM micrograph of a system 3 specimen cured for 3 days.

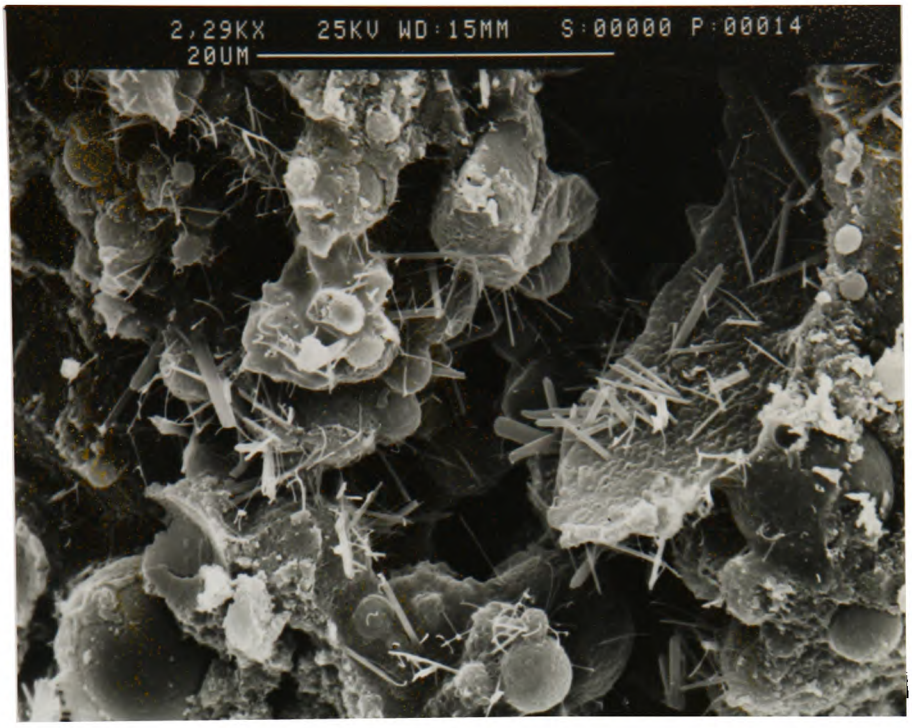


Plate 8.51 SEM micrograph showing network of ettringite needles - system 3 specimen cured for 3 days.

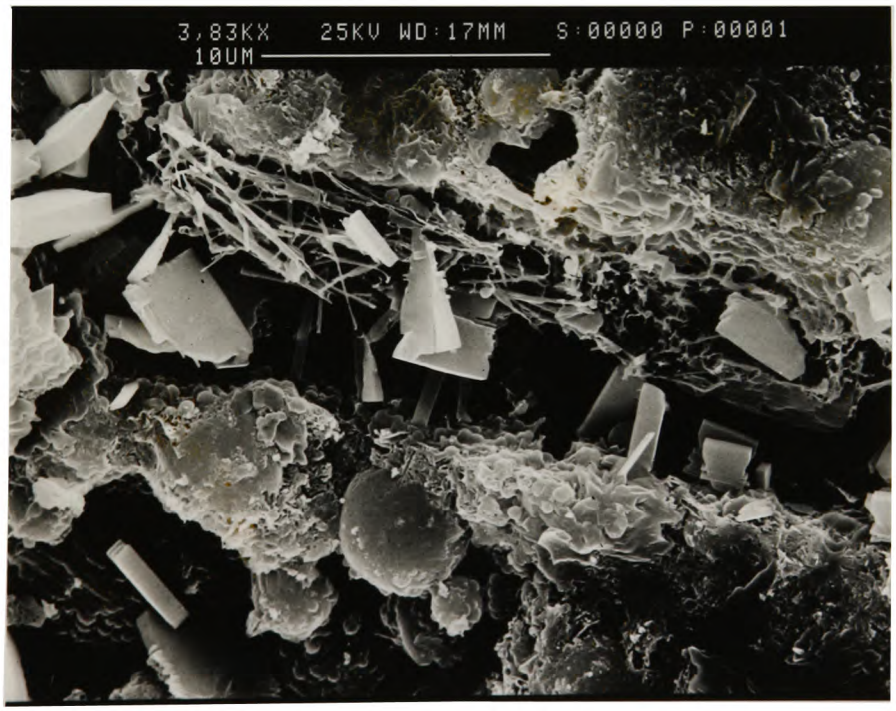


Plate 8.52 SEM micrograph showing gypsum crystals system 3 specimen cured for 7 days.

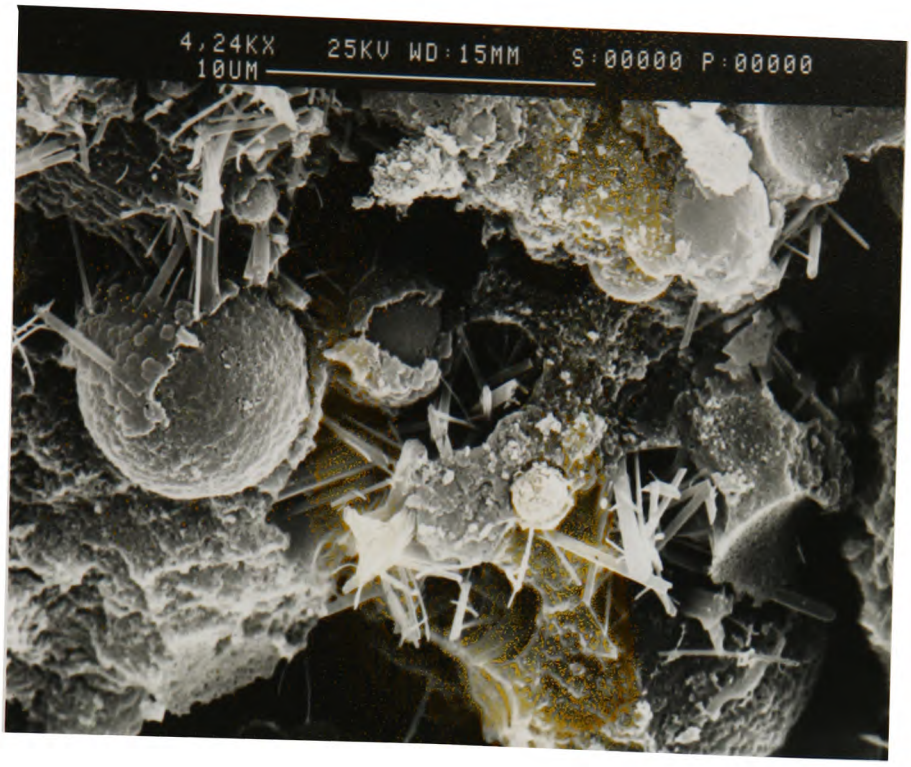


Plate 8.53 SEM micrograph of a system 3 specimen cured for 7 days.

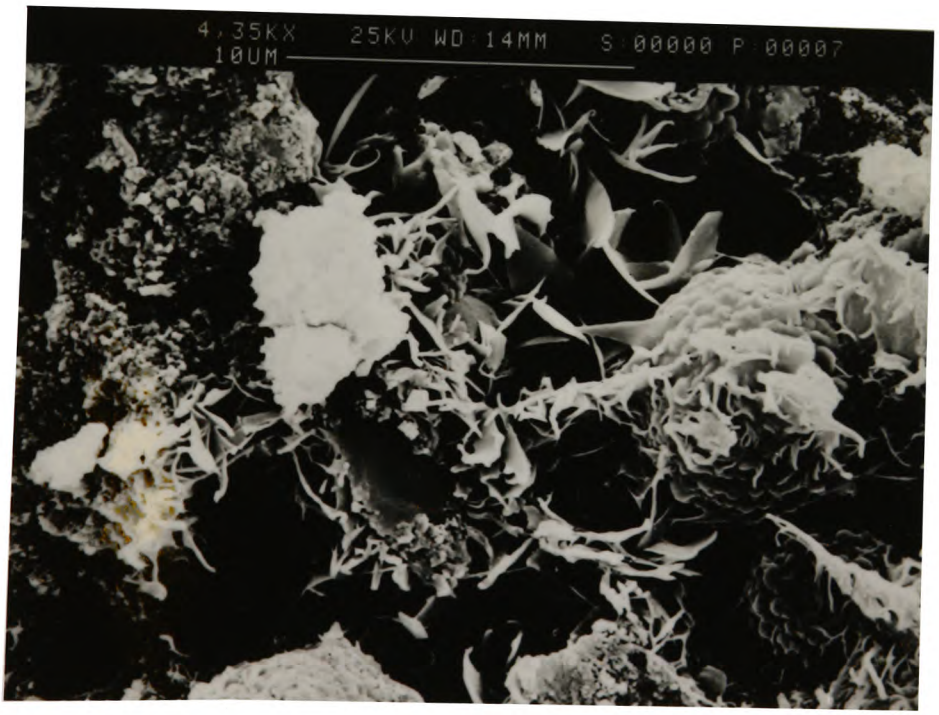
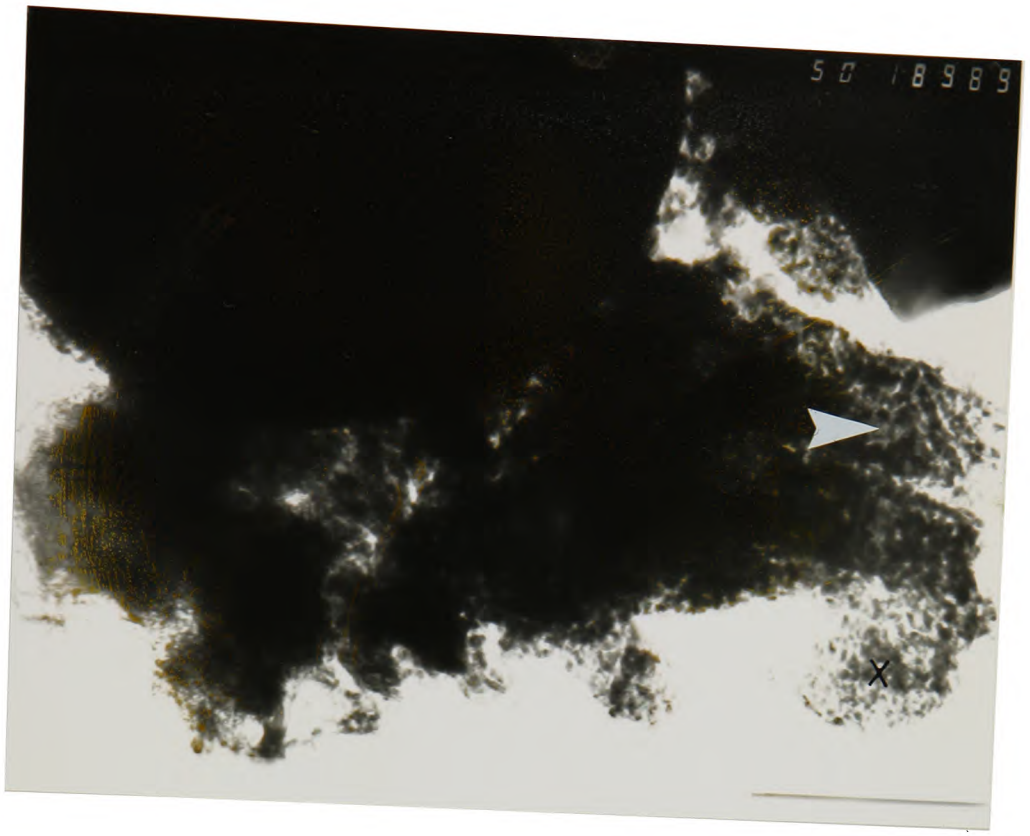


Plate 8.54 SEM micrograph of a system 3 specimen cured for 14 days.

Plate 8.55 SEM micrograph showing foil-like reaction product - system 3 specimen cured for 28 days.



110 nm

EDAX Results

E	%	Ratio to Si
Na	1.02	0.04
Al	27.80	1.07
Si	26.1	-
S	8.1	0.31
K	2.30	0.09
Ca	35.7	1.37
Fe	1.30	0.05

Plate 8.56 TEM micrograph of colloidal product - system 3 specimen cured for 6 hours.

189

EDAX Results

E	%	Ratio to Si
Na	1.12	0.06
Al	29.1	1.46
Si	19.9	-
S	8.53	0.43
K	2.41	0.12
Ca	37.41	1.88
Fe	1.43	0.07

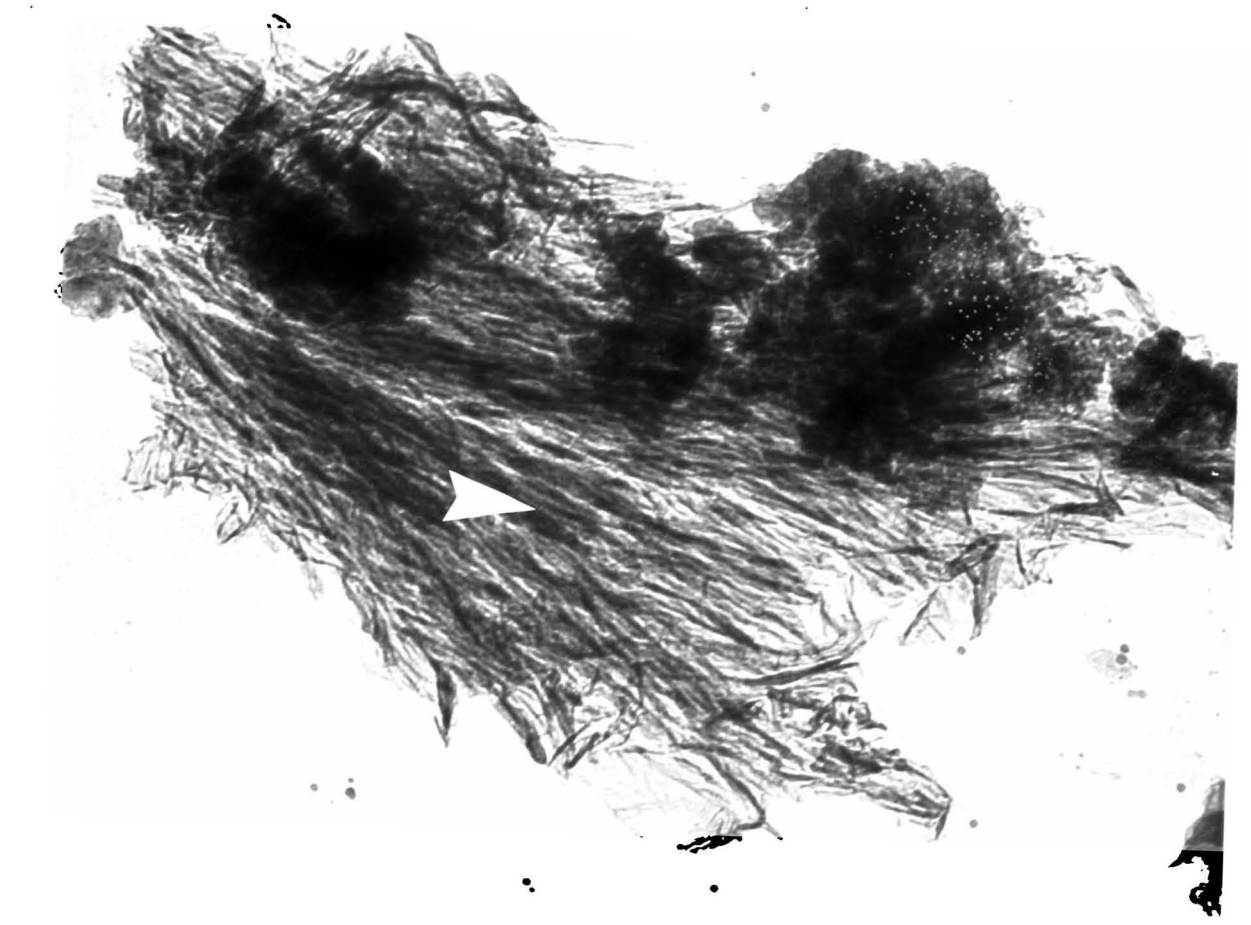
Plate 8.57 TEM micrograph of colloidal product - system 3 specimen cured for 24 hours.

200 nm

EDAX Results

E	%
Na	0.47
Al	3.88
Si	6.84
S	33.45
K	1.23
Ca	51.58
Fe	2.40

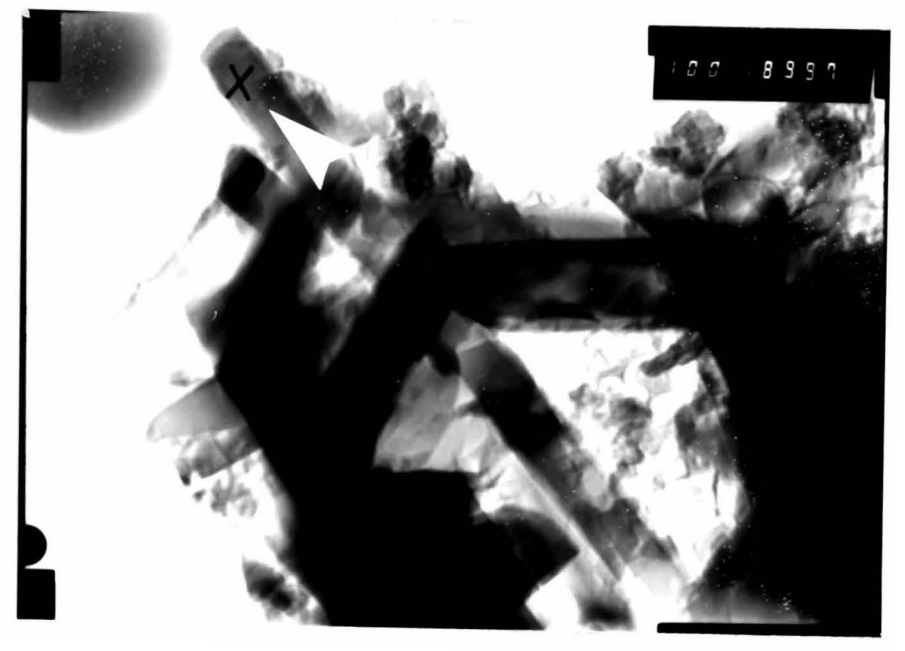
Plate 8.58 TEM micrograph of a gypsum crystal - system 3 specimen cured for 24 hours.



EDAX Results

E	%	Ratio to Si
Na	1.21	0.03
Al	17.26	0.38
Si	45.61	-
S	1.48	0.03
K	1.40	0.03
Ca	30.37	0.67
Fe	2.38	0.05

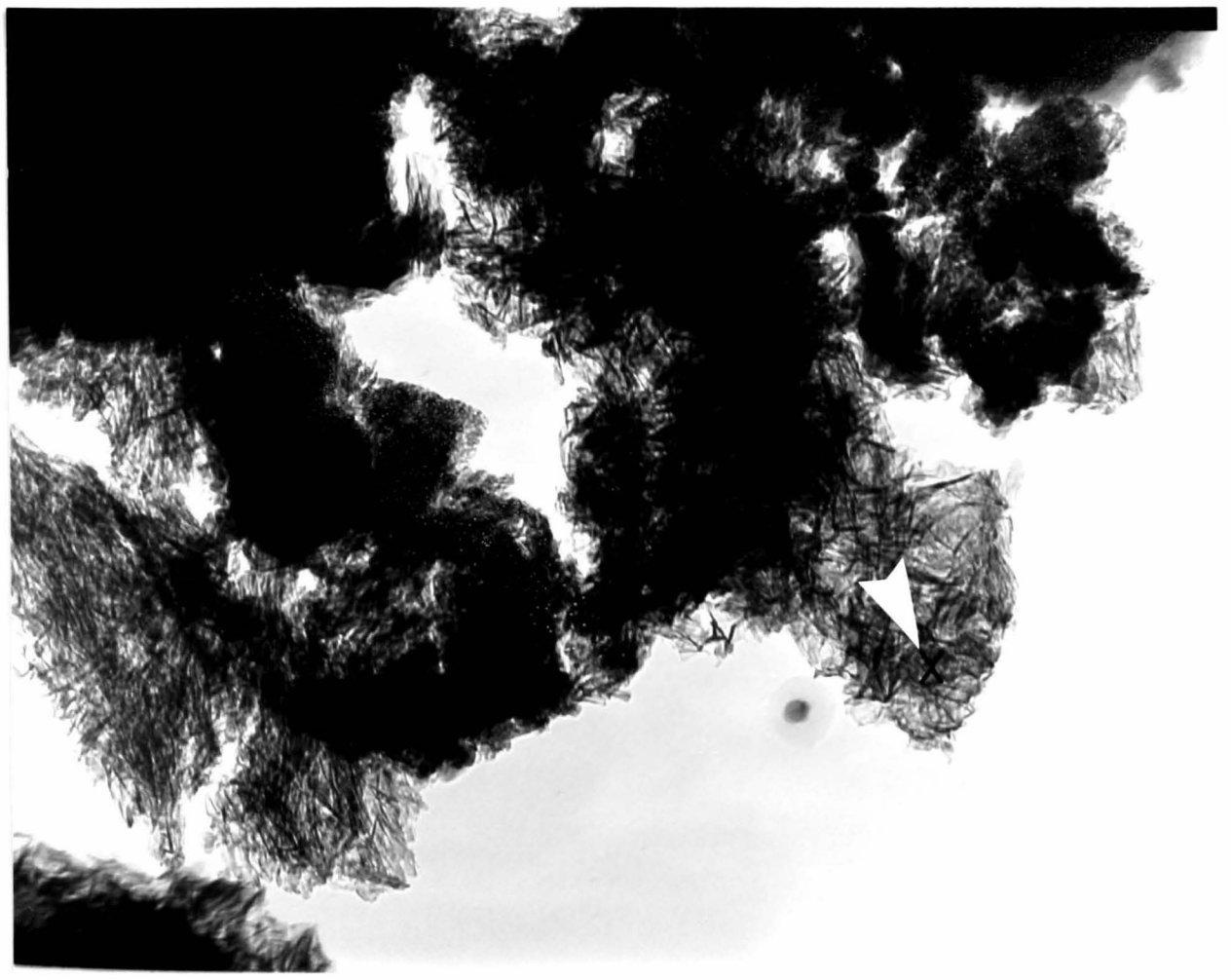
Plate 8.59 TEM micrograph of fibrous gel - system 3 specimen cured for 24 hours.



EDAX Results

E	%	Ratio to Si
Na	-	-
Al	62.84	2.55
Si	24.69	-
S	0.81	0.03
K	1.23	0.05
Ca	8.07	0.33
Fe	2.12	0.09

Plate 8.60 TEM micrograph of mullite crystals - system 3 specimen cured for 24 hours.



EDAX Results

E	%	Ratio to Si
Na	1.12	0.02
Al	17.32	0.38
Si	45.01	-
S	1.93	0.04
K	3.39	0.08
Ca	28.67	0.64
Fe	2.23	0.05

Plate 8.61 TEM micrograph of fibrous gel - system 3 specimen cured for 3 days.

CHAPTER NINE

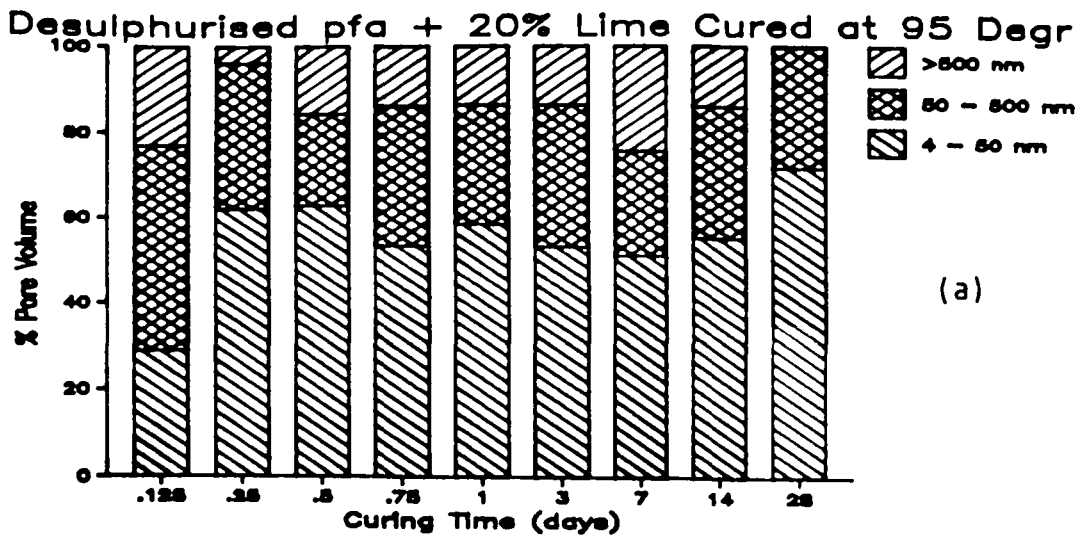
TABLES, FIGURES AND PLATES

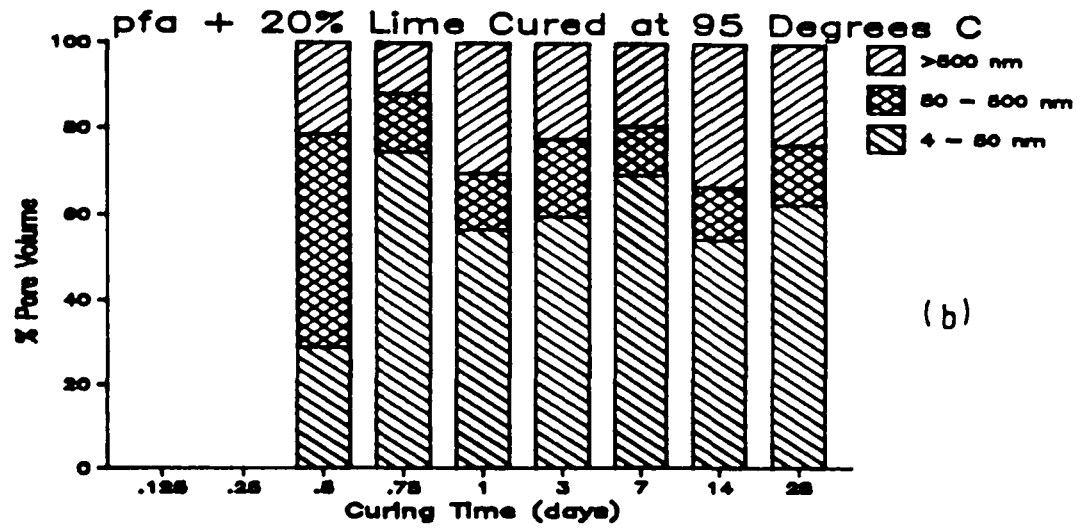
Table 9.1

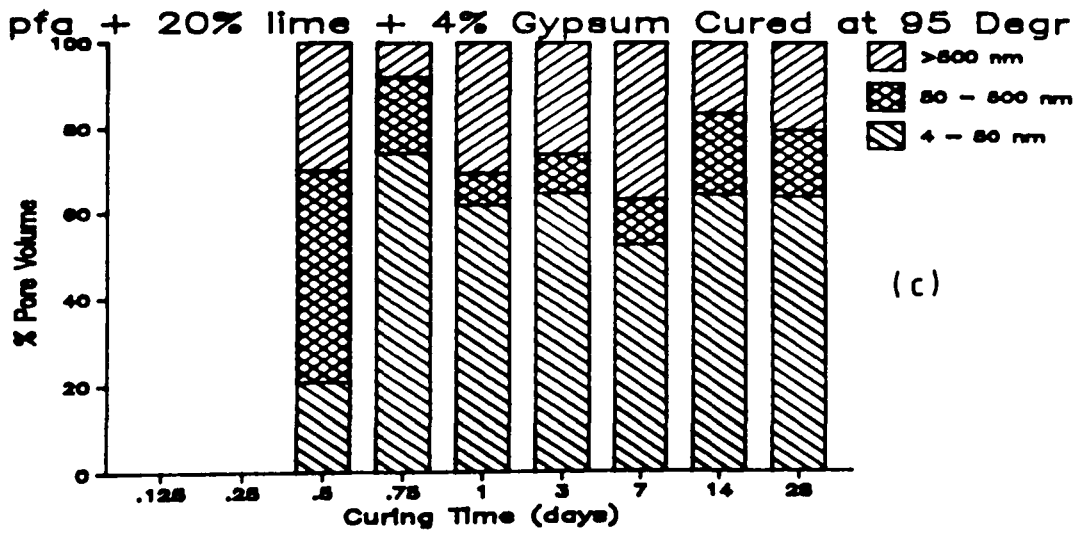
Percentage weight loss and drying shrinkage* for 28 days cured specimen from the four different systems.

System	% Gypsum Addition	% Weight Loss	%Shrinkage
D pfa	- 1.3	12.55	0.058
1	0	15.35	0.077
2	4	14.96	0.251
3	6	15.70	0.558

* - from saturated to oven dried at 95°C.







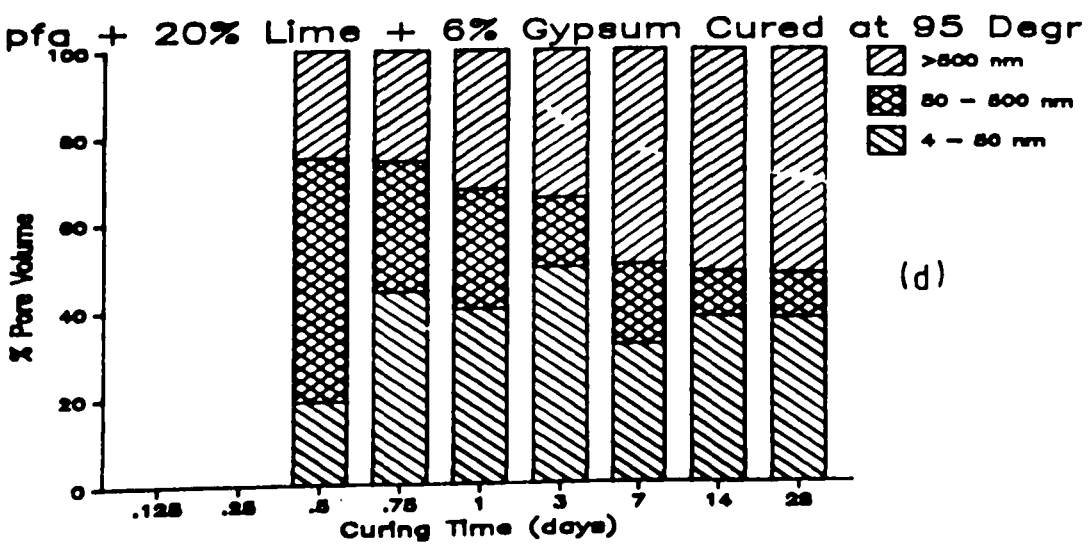
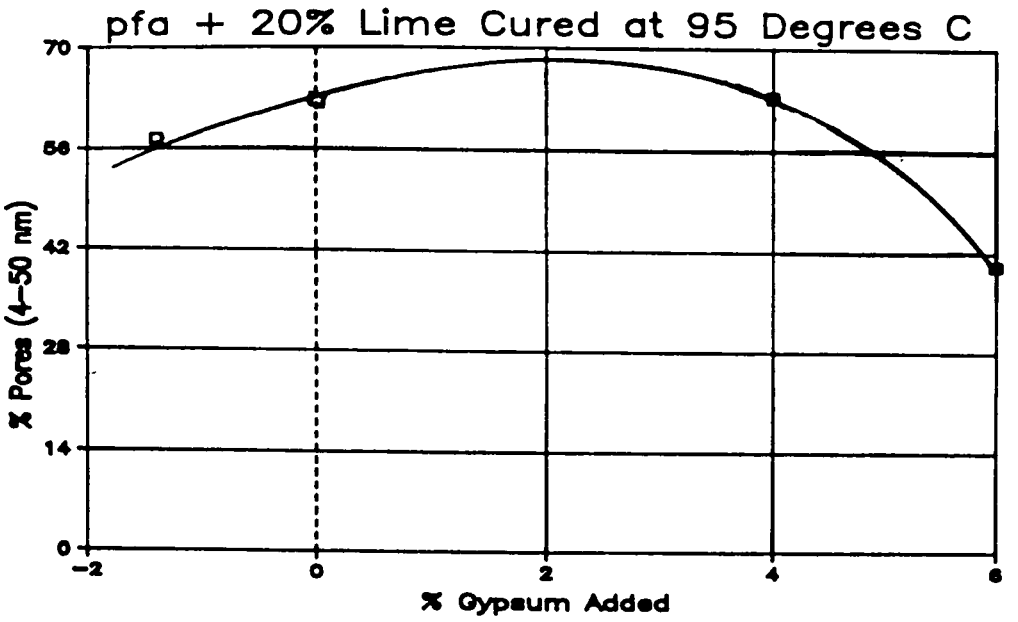
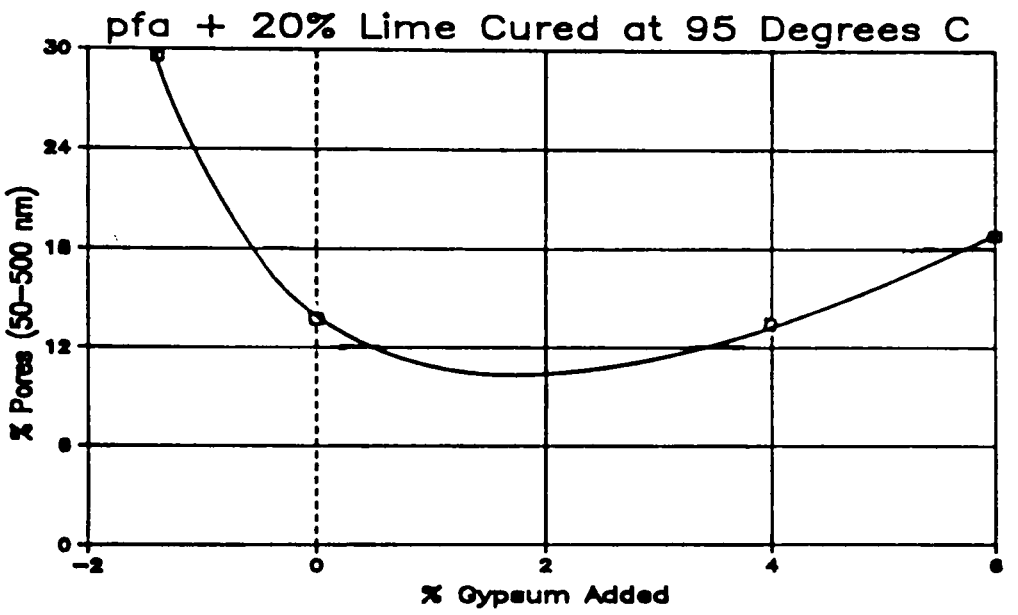


Figure 9.1 Bar charts showing the pore size populations at different curing times in the pore size ranges 4-50 nm, 50-500 nm and › 500 nm for a) the D pfa System, b) System 1, c) system 2 and d) System 3.





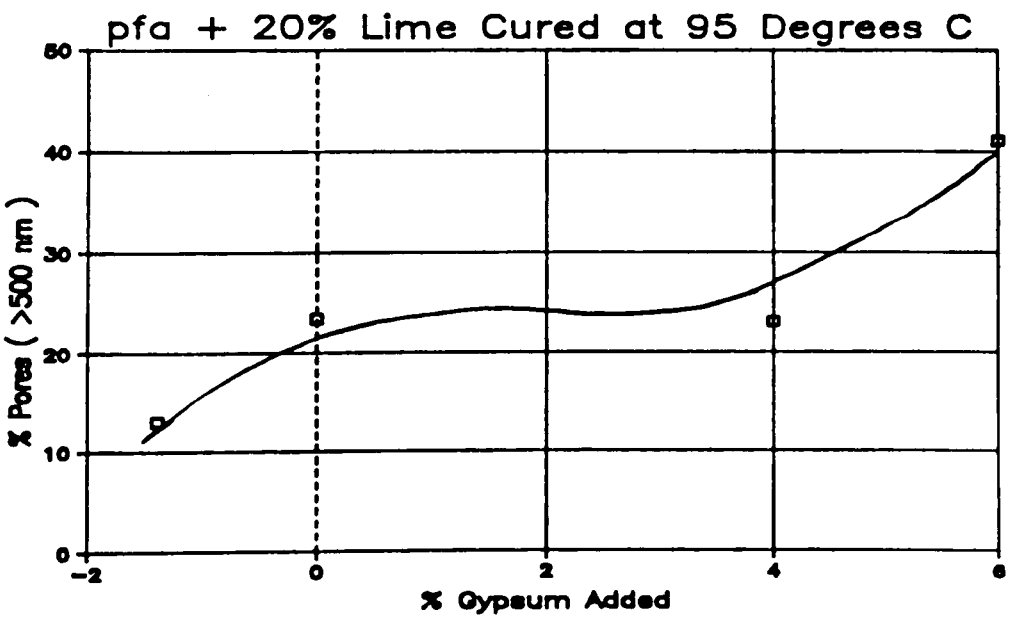


Figure 9.2. Plots of the percentage porosity in each pore size range indicated against gypsum content.

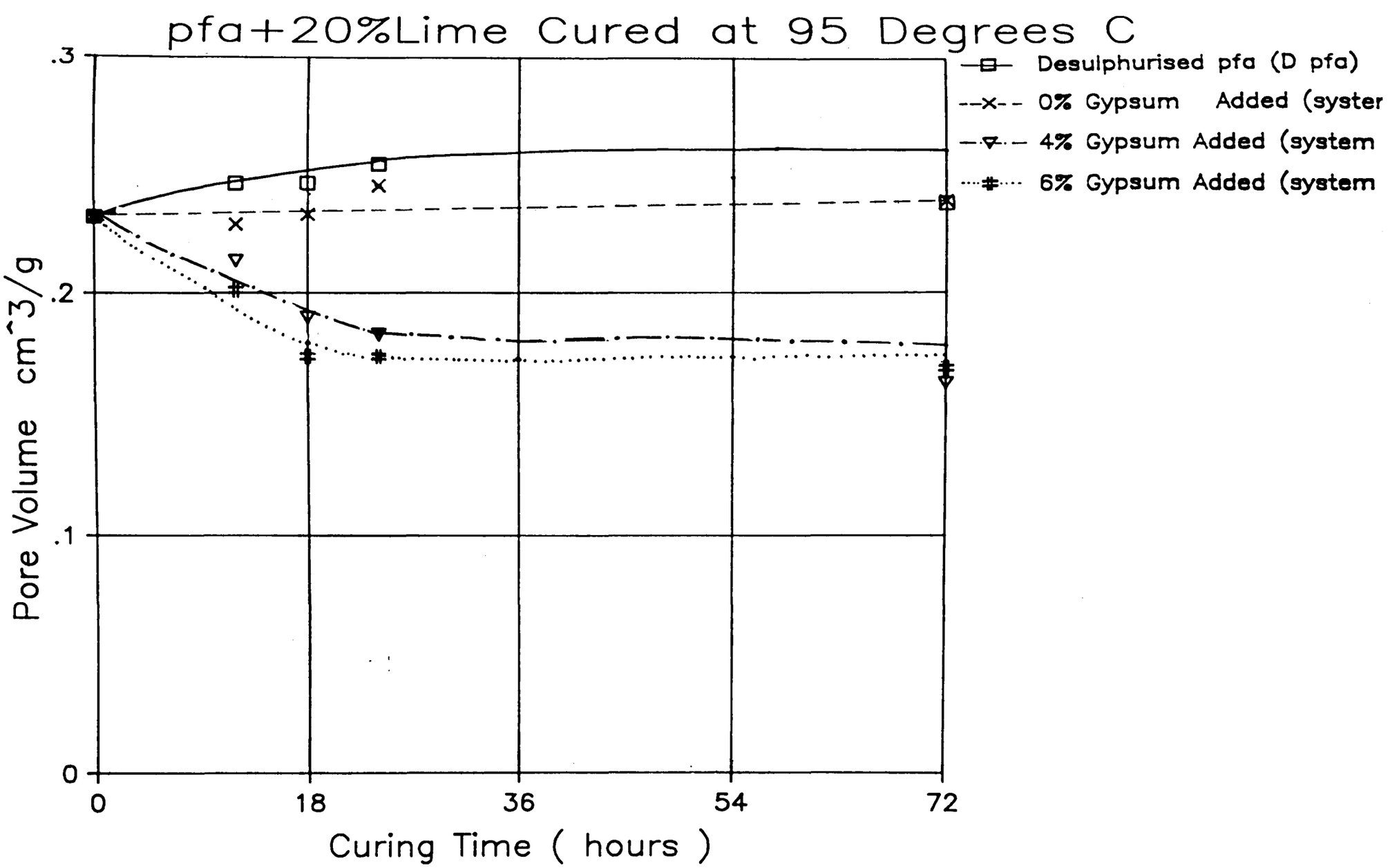


Figure 9.3. Plots of pore volume per unit mass versus curing time for samples cured up to 3 days for the four different systems.

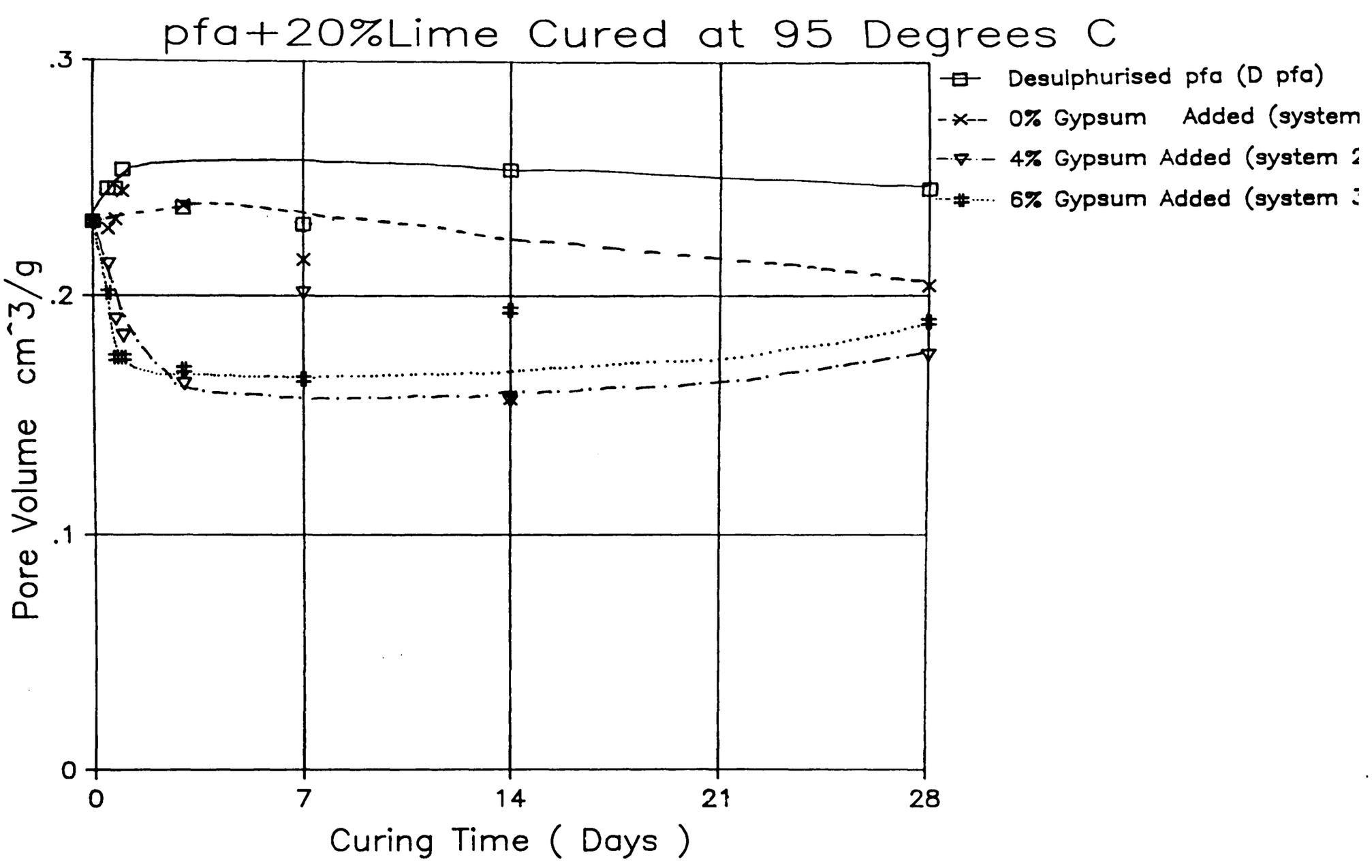


Figure 9.4. Plots of pore volume per unit mass versus curing time for samples cured up to 28 days for the four different systems.

CHAPTER TEN

TABLES, FIGURES AND PLATES

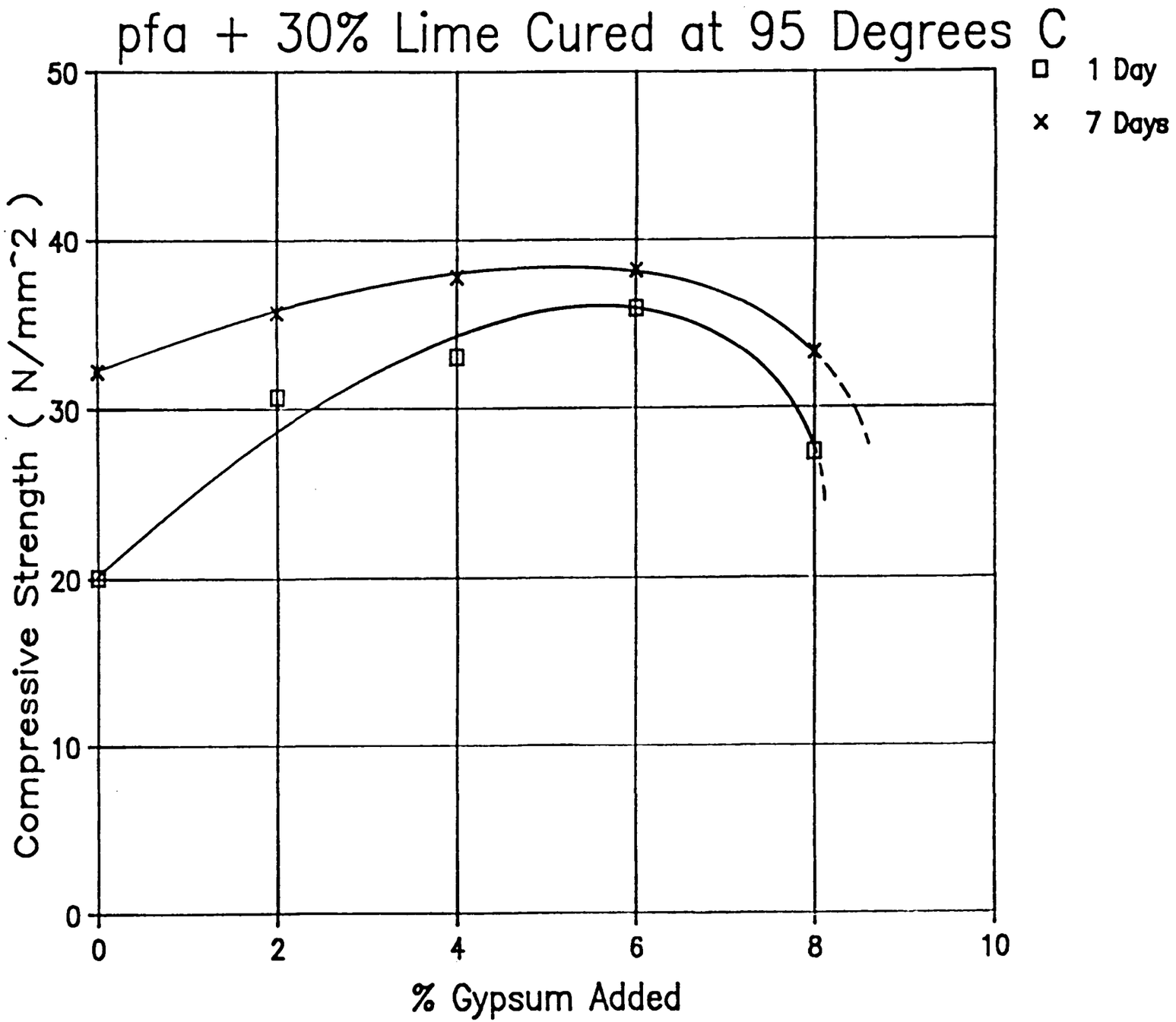


Figure 10.1 Compressive strength versus % gypsum additions - pfa + 30wt.% lime cylinders cured at 95°C and 100% r.h. for 1 and 7 days.

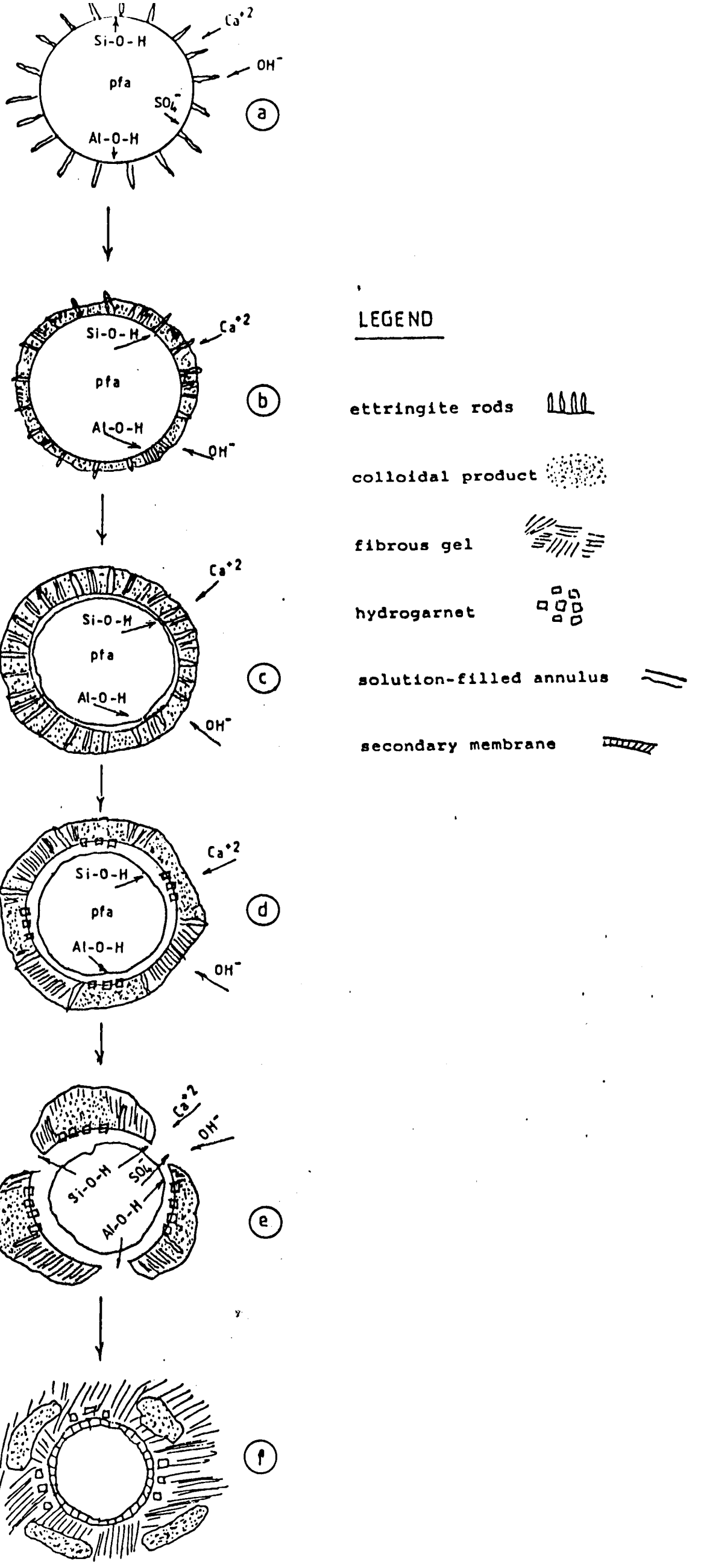


Figure 10.2 Schematic representation of the principal reaction stages for system 1.

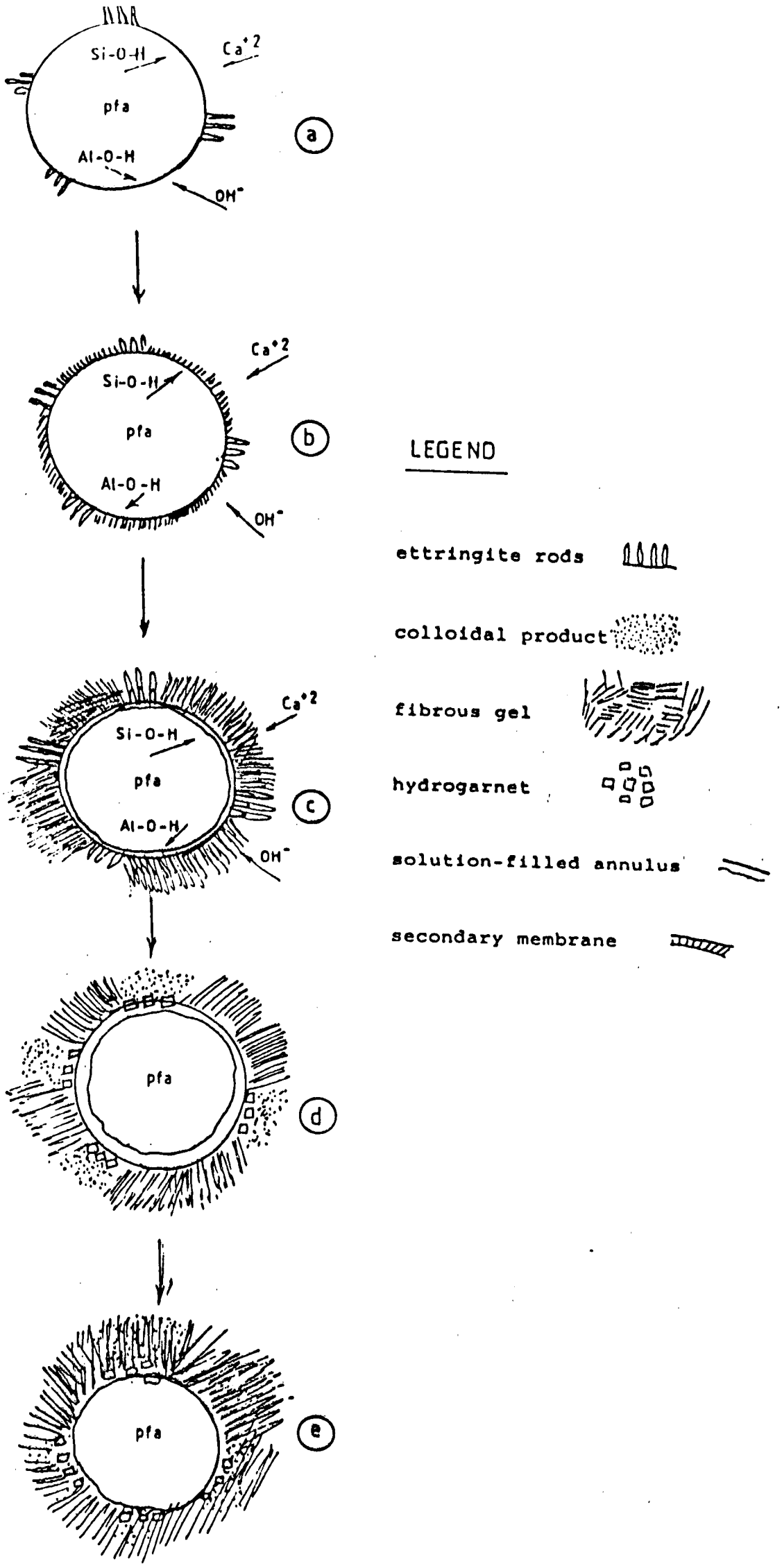


Figure 10.3 Schematic representation of the principal reaction stages for system D pfa.

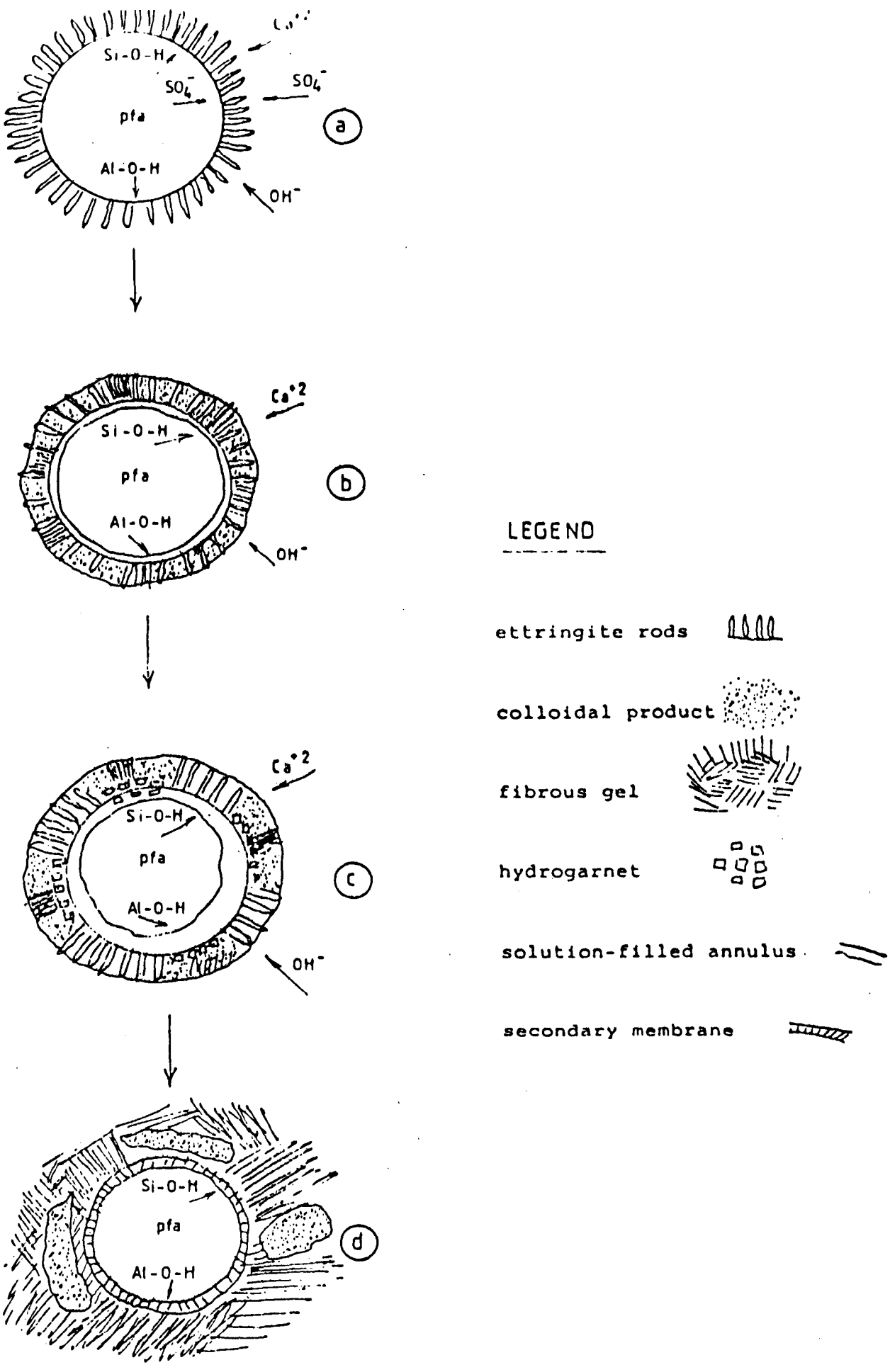


Figure 10.4 Schematic representation of the principal reaction stages for system 2.

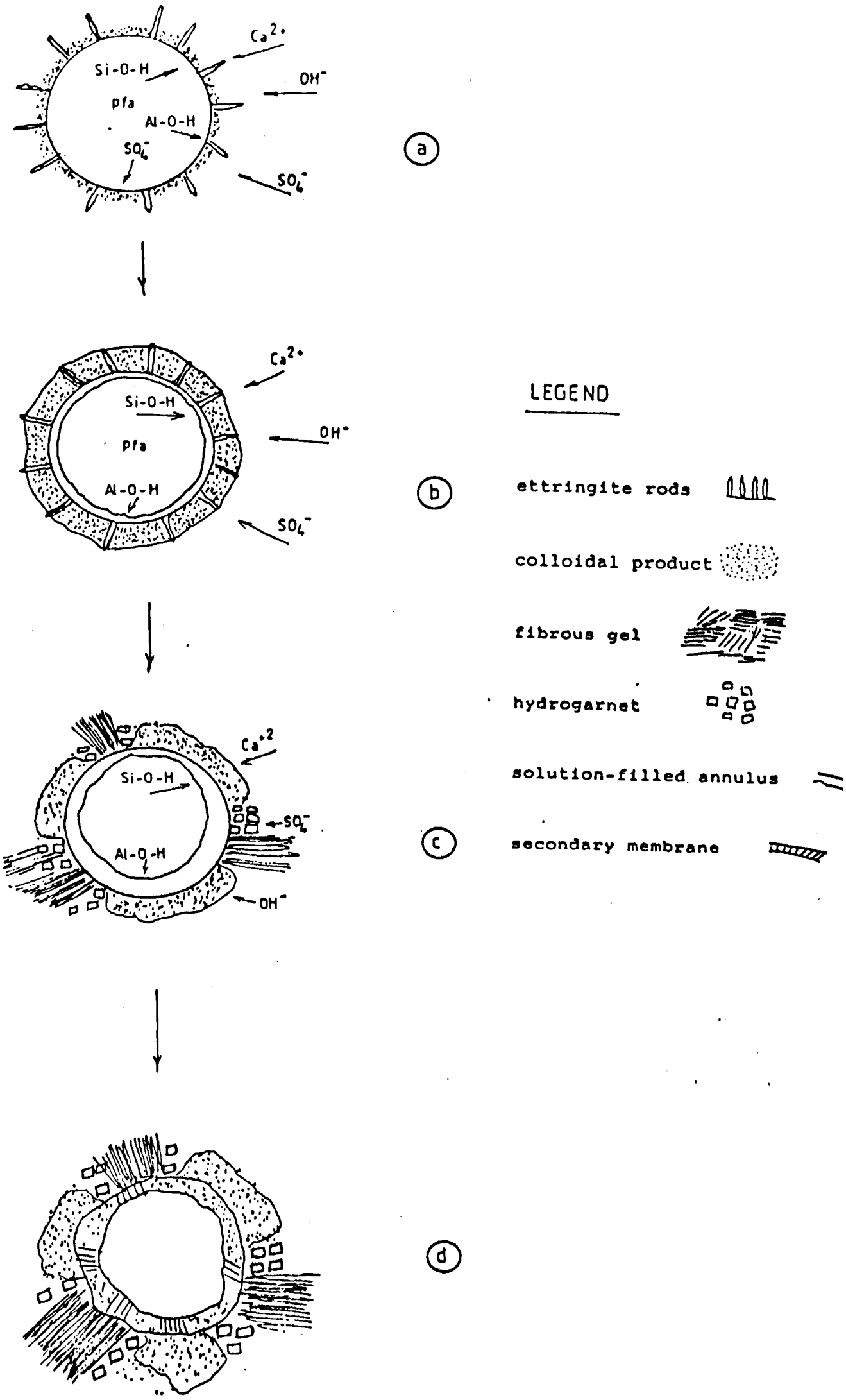


Figure 10.5 Schematic representation of the principal reaction stages for system 3.

APPENDICES

Appendix 1

Method of calculating the weight loss of DTG samples over a specified temperature range

Wi (mg) = Initial weight of the sample,in equillibrium with the initial environment at 20°C,
Wf (mg) = Final weight of the sample, in equilibrium with the final environment at 830-860°C,
dW (mg) = Total weight loss = Wi - Wf,
Wp_t (mg) = Weight of the pfa at temperature t, ti = temperature on ignition, ta = ambient temperature.
W_{CaO} (mg) = Weight of the CaO present.

(1) The weight loss of DTG samples over a certain range of heating temperature can be calculated as follows :

The figure below shows an example of a DTG curve. The total area (vertical slashing) and area A are measured using a computer programme MOSS by digitising the points of the curve into the programme. The weight loss (A (mg)) over area A can then be calculated as :

A (mg) = (Area A / Total Area) x dW eq.1

If for example the weight loss band is identified as that due to free lime dehydroxylation then the weight loss due to the lime only (Wl) over the temperature range 450-550 C is :

Wl (mg) = A (mg) - ($Wp_{450°C}$ - $Wp_{550°C}$) eq.2

The amount of free lime present which produced this particular weight loss is then calculated as follows :

	$Ca(OH)_2$	------	CaO	+	H_2O	
Moler Weight	74	------	56	+	18	eq.3

Since the weight loss is due to the release of water as the free lime transforms into CaO the weight of the lime present in the sample is :

Weight of Free Lime in Sample (mg) = Wl x (74/18) eq.4

(2) The Weight of the pfa and the lime originally added, in the DTG samples can be determined as follows :

The calculations are based on the following assumptions:

(a) At the ignition temperature (830-860°C), the only solid materials remaining in the sample are the oxide components which make up the pfa (Wp_{ti}) plus CaO (W_{CaO}). The CaO will be present due to the decomposition of the added lime and carbonate. The latter will decompose in the following manner :

	$CaCO_3$	------	CaO	+ CO_2	
Moler Weights	100	------	56	+ 44	eq.5

(b) The lime which was present in the DTG samples, was present in exactly the same proportions as in the bulk samples i.e for the "pfa + 20wt.% lime" system the amount of lime present in DTG samples was assumed to be 20wt.% of the weight of the pfa present in those samples :

If the pfa loses k% on ignition (expressed as a percentage of the weight of the pfa) and the carbonate present after curing is c% (expressed as a percentage of the weight of the lime) then the final weight of the DTG sample Wf is given by :

$$Wf = Wp_{ti} + W_{CaO} \text{ from free lime} + W_{CaO} \text{ from carbonate} \qquad \text{eq.6}$$

$$Wf = [Wp_{ta}\,(1-(k/100))] + [Wp_{ta}\,(0.2-\{0.2 \times (c/100)\})] \times (56/74) + [Wp_{ta} \times 0.2 \times (c/100) \times (56/100)] \qquad \text{eq.7}$$

If k is known from previous DTG tests on the pfa and c is determined from DTG tests on the cured specimens then the initial weight of the pfa (Wp_{ta}) present in the sample can be determined by determining the final ignited weight (Wf) of the cured sample.

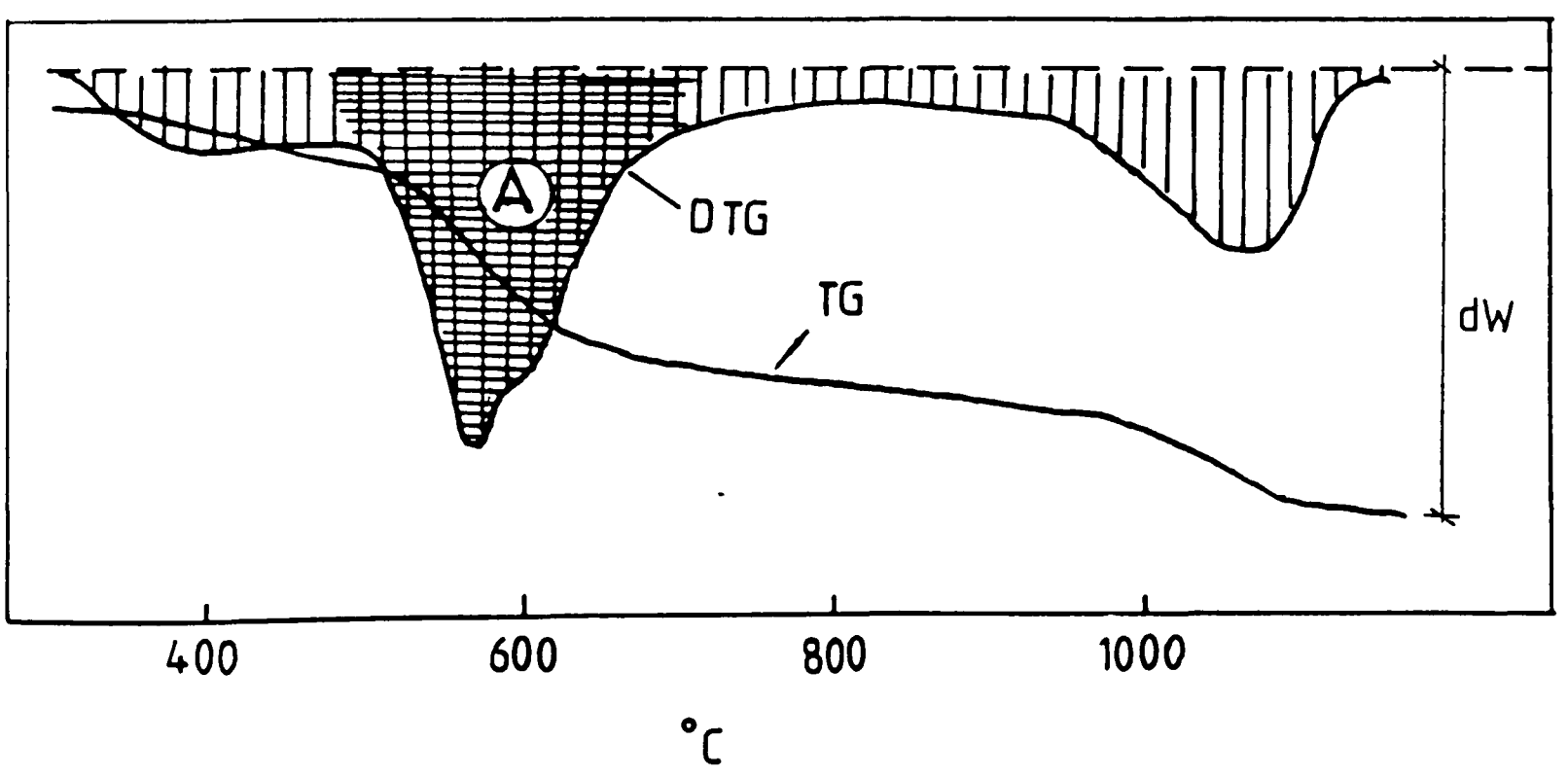
(3) When g% of gypsum (expressed as a percentage of the weight of the pfa) has also been added to the pfa-lime mixes the initial weight of the pfa (Wp_{ta}) can be calculated using the same assumptions and method as in (2). It is assumed that gypsum decomposes in the DTG test in the following manner :

	$CaSO_4.2H_2O$	-----	$CaSO_4$ +	$2H_2O$	
Moler Weights	172	-----	136 +	36	eq.8

So equation (7) can be modified as below :

$$Wf = [Wp_{ta} (1-(k/100))] + [Wp_{ta} (0.2-\{0.2 \times (c/100)\})] \times (56/74) + [Wp_{ta} (g/100) \times (136/172)] \quad \text{eq.9}$$

Therefore by knowing Wf, k%, c% and g%, the original weight of the pfa (Wp_{ta}) can be calculated.



Appendix 2

General Characteristics Of Calcium Sulphate

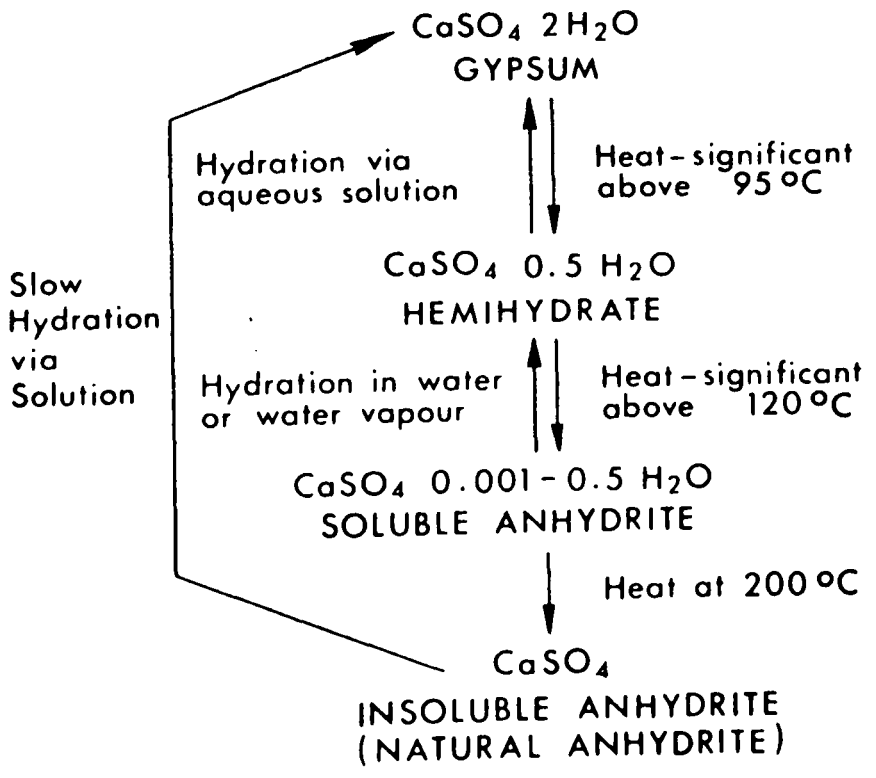
1. Solubility of calcium sulphate in water [95] :

temperature °C	grams $CaSO_4$ per 100 cc solution
0	0.1759
10	0.1928
18	0.2016
25	0.2080
30	0.2090
35	0.2090
40	0.2097
55	0.2009
75	0.1847
100	0.1619

2. Solubility of calcium sulphate in aqueous solutions of HCl [95] :

grams acid per 100 cc solution	grams $CaSO_4$ per 100 cc solution	
	at 25°C	at 102°C
0	0.208	0.16
1	0.720	1.38
2	1.02	2.38
3	1.25	3.20
4	1.42	3.64
6	1.65	4.65

3. Calcium sulphate - water system [75] :



4. Solubilities of various sulphates [75] :

Compound	Formula	Solubility (g/l)
Hemihydrate	$CaSO_4\ \frac{1}{2}H_2O$	2.4
Soluble anhydrite	$CaSO_4\ 0.001\text{-}\frac{1}{2}H_2O$	≈6
Insoluble anhydrite	$CaSO_4$	≈6
Syngenite	$K_2Ca(SO_4)_2H_2O$	2.5
Potassium sulphate	K_2SO_4	136

Appendix 3

Method of calculating various parameters related to the compaction studies of pfa and lime mixtures

Assume,
Wp = Weight of the pfa only
Wl = Weight of the Added Lime only
Ww = Weight of the water only
Ws = Weight of the solid materials (Wp + Wl)
Wt = Total Weight of the mix (Wp + Wl + Ww)
Vp = Volume of the pfa only
Vl = Volume of the added lime only
Vw = Volume of the water only
Vs = Volume of the solid materials (Vp + Vl)
Va = Volume of the air present
Vt = Total volume of the mix (Vp + Vl + Vw + Va)
Dp = Density of the pfa (taken as 2.38 g/cm3)
Dl = Density of the lime (taken as 2.24 g/cm3)
Dw = Density of water (taken as 1.00 g/cm3)

(1) Relationship between moisture content expressed in terms of weight of total solids and moisture content expressed in terms of weight of pfa

If the optimum moisture content for pfa + 20wt.% added lime expressed in terms of the weight of total solids is found experimentally to be 25.2% the optimum moisture content expressed in terms of the weight of pfa can be calculated as follows,

$$Ww/Ws = Ww/(Wp + Wl)$$
$$= Ww/(Wp + 0.2\ Wp)$$
$$= Ww/(1.2\ Wp)$$
$$Ww/Wp = 1.2\ (Ww/Ws) \qquad \text{eq.1}$$

therefore if $Ww/Ws = 0.252$,

$$Ww/Wp = (1.2 \times 0.252) \times 100 = 30.2\%$$

(2) Conversion of total solids dry density to pfa dry density,

$$\text{Total solids dry density} = Ws/Vt$$
$$= (Wp + Wl)/Vt$$

so if for example lime is added at 20wt.%,

$$\text{Total Dry Density} = (Wp + 0.2\ Wp)/Vt$$
$$= 1.2\ Wp/Vt$$

therefore pfa dry density,

$$Wp/Vt = \text{Total solids dry Density}/1.2 \qquad \text{eq.2}$$

(3) Determination of theoretical total solids dry density assuming no air voids are present (Va=0),

Total solids dry density = Ws/Vt

$$= (Wp + Wl)/(Vp + Vl + Vw)$$
$$= (Wp + Wl)/[(Wp/2.37) + (Wl/2.24) + (Ww/1.00) \quad \text{eq.3}$$

Therefore if the weight of the pfa, the weight of the added lime, and the moisture content are known the theoretical total solids dry density can be calculated as in the following example :

Assume a pfa + 20wt.% lime mix with a water/solids ratio of 0.252 (see Table 5.1) which is equivalent to a water/pfa ratio of 0.302. Hence

Wl = 0.2 Wp and Ww = 0.302 Wp
Therefore the calculated total solids dry density,
Ws/Vt = 1.2 Wp/ Wp[(1/2.38)+(0.2/2.24)+(1/0.302)]
= 1.2/0.813
= 1.476

(4) Determination of the percentage air voids;

Fraction of air voids = 1 - (Vs+Vw)/(Vs+Vw+Va)
= 1 - M(Vs+Vw)/M(Vs+Vw+Va)
= 1 - pm/pc eq.4

Where pm and pc are respectively the measured and calculated densities. Therefore for a pfa + 20wt.% lime mix at a water/solids ratio of 0.252 (see Table 5.1) the fraction of air voids is,

= 1 - 1.37/1.476
= 0.0718 or 7.18%

pfa + 20% Lime Cured at 50 °C																			
Hydrogarnet (H)																			
Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
0·503	s					–	–	–	–	–	–	–	–	–	–	–	–	–	–
0·436	s					–	–	–	–	–	–	–	–	–	–	–	–	–	–
0·329	m					–	–	–	–	–	–	–	–	–	–	–	–	–	–
0·308	s					–	–	–	–	–	–	–	–	–	–	–	–	–	–
0·276	m					–	–	–	–	–	–	–	–	–	–	–	–	–	–
0·251	s					–	–	–	–	–	–	–	–	–	–	–	–	–	–
0·242	m					–	–	–	–	–	–	–	–	–	–	–	–	–	–
0·225	m					–	–	–	–	–	–	–	–	–	–	–	–	–	–
0·200	ms					–	–	–	–	–	–	–	–	–	–	–	–	–	–
Lime (L).																			
0·49	vs					0·488	s	0·484	ms	0·491	ms	0·484	w	0·487	m	0·489	w	–	–
0·311	m					0·309	w	0·302	w	0·309	w	0·310	w	0·308	w	0·316	w	–	–
0·262	vvs					0·262	s	0·261	s	0·261	s	0·261	ms	0·261	ms	0·261	m	0·261	w
0·192	ms					0·192	vw	0·191	w	0·192	vw	0·191	vw	0·191	vw	0·191	vw	–	–
0·179	ms					0·179	vw	0·178	w	0·179	vw	0·179	vw	0·179	vw	–	–	–	–
0·168	m					0·168	vw	–	–	0·167	vw	–	–	–	–	–	–	–	–

Appendix 4 XRD data for mixtures of pfa + 20wt.% lime cured at 50°C and 100% r.h. up to 28 days.

d = d specings (nm), I = peak intensity, w = weak, s = strong, m = mild, v = very.

pfa + 20% Lime Cured at 50 °C

Ettringite (E)

Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
0·973	vvs					0·955	w	0·952	w	0·955	w	0·96	w	0·952	w	0·969	w	—	—
0·561	vs					0·556	vw	0·557	vw	0·557	vw	0·553	vw	0·556	vw	0·559	vw	—	—
0·498	m					—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·464	ms					—	—	0·464	w	0·468	vw	0·465	vw	—	—	—	—	—	—
0·388	s					0·385	w	0·386	w	0·386	w	0·385	w	0·385	w	0·385	w	—	—
0·348	m					—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·277	ms					0·275	vw	0·274	vw	0·276	vw	0·276	w	0·276	w	0·275	w	—	—
0·256	s					0·253	w	0·253	w	0·254	w	0·253	w	0·253	w	0·253	w	—	—
0·220	s					0·220	vw	0·219	vw	0·220	w	0·219	vw	0·219	w	0·220	vw	—	—
Monosulphate (Mo)																			
0·892	vs					—	—	—	—	—	—	—	—	—	—	—	—	0·825	ms
0·446	s					—	—	—	—	—	—	—	—	—	—	—	—	0·416	vvs
0·399	s					—	—	—	—	—	—	0·399	w	0·399	w	—	—	0·402	s
0·287	vs					—	—	—	—	—	—	—	—	—	—	—	—	0·286	s
0·245	s					—	—	—	—	—	—	0·245	vw	—	—	—	—	0·242	ms
0·241	m					—	—	—	—	—	—	0·241	vw	0·241	vw	—	—	0·241	w

Appendix 4 continued...

pfa + 20% Lime Cured at 50 °C

Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
Calcite (Ca)																			
0·303	vvs					—	—	0·301	w	0·301	w	0·302	ms	—	—	—	—	—	—
0·249	m					—	—	—	—	—	—	0·248	vw	—	—	—	—	—	—
0·228	s					0·228	vw	0·228	vw	0·228	vw	0·227	vw	0·228	vw	0·228	vw	0·228	vw
0·209	s					—	—	—	—	—	—	—	—	—	—	—	—	—	—
Mullite (M)																			
0·539	vs					0·536	w	0·533	vw	0·538	vw	0·537	w	0·535	w	0·538	w	0·536	w
0·342	vs					0·341	ms	0·341	w	0·342	w	0·341	ms	0·341	w	—	—	0·341	w
0·339	vvs					0·338	ms	0·338	ms	0·338	ms	0·338	ms	0338	ms	0·339	ms	0·339	ms
0·269	vs					0·268	w	0·268	w	0·269	w	0·268	w	0·268	w	0·268	w	0·268	w
0·254	vs					0·253	w	0·253	w	0·254	w	0·253	w	0·253	w	0·253	w	0 253	w
0·220	vs					0·220	vw	0·219	vw	0·220	vw	0·219	vw	0·219	w	0·220	w	0·220	w

Appendix 4 continued...

pfa + 20% Lime Cured at 50 °C

Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
Quartz (Q)																			
0·426	s					0·426	m	0·422	m	—	—	0·424	m	0·425	m	0·427	m	—	—
0·334	vvs					0·334	s	0·333	s	0·334	s	0·333	s	0·333	s	0·333	s	0·334	s
Haemetite (Ha)																			
0·367	s					—	—	—	—	0·364	w	—	—	—	—	—	—	—	—
0·269	vvs					0·268	w			0·269	w	0·268	w	0·288	w	0·268	w	0·268	w
0·251	vs					0·250	w	0·250	w	0·251	w	0·250	w	0·251	w	0·251	w	0·251	w
0·220	s					0·220	vw	0·219	vw	0·220	vw	0·219	vw	0·219	w	0·220	w	0·220	vw
Magnetite (Mg)																			
0·297	vs					0·293	w	0·293	vw	0·295	w	0·294	w	0·293	w	—	—	0·295	w
0·253	vvs					—	—	—	—	—	—	—	—	—	—	—	—	—	—

Appendix 4 continued...

Pfa + 20% Lime Cured at 75 °C																			
Hydrogarnet (H)																			
Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
0·503	s					—	—	0·500	w	0·501	vw	0·501	vw	0·501	vw	0·502	vw	0·502	vw
0·436	s					—	—	—	—	0·432	w	0·435	vw	0·434	w	0·437	w	0·434	w
0·329	m					—	—	0·329	w	—	—	0·328	w	0·329	w	0·328	w	0·300	w
0·308	m					—	—	—	—	0·308	w	0·309	w	0·308	w	0·307	ms	0·307	ms
0·276	s					—	—	0·276	w	0·275	w	0·274	w	0·275	w	0·270	ms	0·275	ms
0·251	m					—	—	0·251	w	0·251	w	0·251	w	0·251	w	0·251	w	0·251	w
0·242	m					—	—	—	—	—	—	—	—	—	—	0·242	vw	0·248	vw
0·225	m					—	—	—	—	0·225	vw	0·225	vw	0·224	vw	0·224	vw	0·224	vw
0·200	ms					—	—	0·200	vw	0·199	vw	0·199	vw	0·199	vw	0·199	vw	0·200	vw
Lime (L).																			
0·49	vs					0·490	s	0·486	ms	0·486	w	0·486	w	0·487	vw	—	—	—	—
0·311	m					0·309	w	0·309	w	0·308	w	0·309	w	—	—	—	—	—	—
0·262	vvs					0·262	vvs	0·262	s	0·261	ms	0·261	w	0·217	w	—	—	—	—
0·192	ms					0·192	w	0·192	vw	0·192	vw	—	—	—	—	—	—	—	—
0·179	ms					0·178	w	0·179	vw	0·179	vw	0·179	vw	—	—	—	—	—	—
0·168	m					0·168	vw	—	—	—	—	—	—	—	—	—	—	—	—

Appendix 5 XRD data for mixtures of pfa + 20wt.% lime cured at 75°C and 100% r.h. up to 28 days.

d = d specings (nm), I = peak intensity, w = weak, s = strong, m = mild, v = very.

pfa + 20% Lime Cured at 75°C

Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
Ettringite (E)																			
0·973	vvs					0·960	w	0·960	w	—	-	—	—	—	—	—	—	—	—
0·561	vs					0·557	vw	0·554	vw	-	-	—	—	—	—	—	—	—	—
0·498	m					—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·469	ms					0·469	w	0·464	w	0·464	vw	—	—	—	—	—	—	—	—
0·388	s					0·387	w	0·385	w	0·386	w	—	—	—	—	—	—	—	—
0·348	m					0·326	w	—	—	—	—	-	—	—	—	—	—	—	—
0·277	ms					0·276	vw	0·276	w	0·275	w	0·274	w	0·275	w	—	—	—	—
0·256	s					0·254	w	0·254	w	0·253	w	—	—	—	—	—	—	—	—
0·220	s					0·220	w	0·220	w	0·220	vw	0·220	vw	0·220	vw	—	—	—	—
Calcite (Ca)																			
0·303	vvs					—	—	0·302	w	0·302	w	0·303	w	0·302	w	—	—	—	—
0·249	m					—	—	—	-	-	—	—	—	—	—	—	—	—	—
0·228	s					—	—	—	—	0·229	vw	0·229	vw	0·228	vw	—	—	—	—
0·209	s																		
0;191	ms																		
0·187	ms																		
C-S-H																			
0·307	vs							0·302	w	0·302	w	0·303	w	0·302	w				
0·280	s					0·276	vw	0·276	w	0·275	w	0·274	w	0·275	w				
0·183	s																		

Appendix 5 continued...

pfa + 20% Lime Cured at 75°C																			
Mullite (M)																			
Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
0·539	vs					0·537	w	0·535	w	0·533	vw	0·536	w	0·536	w	0·539	w	0·536	w
0·342	vs					0·341	ms	0·341	ms	0·341	w	0·341	w	0·340	ms	0·343	ms	0·341	ms
0·339	vvs					0·339	ms	0·338	ms	0·338	ms	0·338	ms	0·338	ms	0·339	ms	0·339	ms
0·269	vs					0·269	w	0·269	w	0·269	w	0·269	w	0·269	w	0·268	w	0·269	w
0·254	vs					0·254	w	0·254	w	0·253	vw	0·254	w	0·253	w	0·253	w	0·254	w
0·220	vs					0·220	w	0·220	w	0·220	vw	0·220	w	0·220	vw	0·220	vw	0·220	vw
Quartz (Q)																			
0·426	s					0·426	w	—	—	0·424	w	0·426	w	0·425	w	0·426	w	0·426	w
0·334	vvs					0·334	s	0·334	s	0·333	s	0·334	s	0·334	s	0·334	s	0·334	s
Haemetite (Ha)																			
0·367	s					—	—	—	—	—	—	0·368	w	—	—	—	—	—	—
0·269	vvs					0·269	w	0·269	w	0·269	w	0·269	w	0·269	w	0·268	w	0·269	w
0·251	vs					0·251	vw	0·251	w	0·251	w	0·251	w	0·251	w	0·251	w	0·251	w
0·220	s					0·220	vw	0·220	w	0·220	vw	0·220	w	0·202	vw	0·220	vw	0·220	vw
Magnetite (Mg)																			
0·297	vs					0·295	w	0·294	w	0·294	w	0·294	w	0·293	w	0·292	w	0·294	w
0·253	vvs					—	—	—	—	—	—	—	—	—	—	—	—	—	—

Appendix 5 continued...

pfa + 20% Lime Cured at 95°C																			
Hydrogarnet (H)																			
Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
0·503	s			–	–	0·505	w	0·506	w	0·504	w	0·503	w	0·503	w	0·504	w	0·505	w
0·436	s			–	–	0·435	w	0·437	w	0·436	w	0·436	w	0·435	w	0·436	w	0·436	w
0·329	m			–	–	0·329	w	0·332	w	0·329	w	0·329	w	0·329	w	0·329	w	–	–
0·308	m			–	–	0·308	w	0·309	w	0·308	w	0·308	ms	0·308	w	0·308	w	0·308	w
0·276	s			–	–	0·276	w	0·277	w	0·276	w	0·276	ms	0·276	w	0·276	w	0·276	w
0·251	m			–	–	–	–	0·252	w	–	–	0·251	w	0·252	w	0·251	w	0·251	w
0·242	m			–	–	–	–	–	–	–	–	0·242	vw	–	–	0·242	vw	–	–
0·225	m			–	–	–	–	–	–	–	–	0·225	w	–	–	0·225	w	0·225	vw
0·200	ms			–	–	–	–	–	–	–	–	0·205	vw	–	–	0·200	w	0·200	vw
Lime (L).																			
0·49	vs			–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–
0·311	m			–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–
0·262	vvs			–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–
0·192	ms			0·192	vw	–	–	–	–	–	–	–	–	–	–	–	–	–	–
0·179	ms			–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–
0·168	m			–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–

Appendix 6 XRD data for mixtures of pfa + 20wt.% lime cured at 95°C and 100% r.h. up to 28 days.

d = d specings (nm), I = peak intensity, w = weak, s = strong, m = mild, v = very.

pfa + 20% Lime Cured at 95°C

Ettringite (E)

Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
0·973	vvs			0·976	w	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·561	vs			0·560	vw	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·498	m			0·491	w	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·469	ms			0·469	vw	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·388	s			0·386	ms	0·386	w	0·386	w	0·385	w	—	—	—	—	—	—	—	—
0·348	m			—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·277	ms			—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·256	s			0·263	w	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·220	s			0·221	w	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Calcite (Cc)																			
0·303	vvs			0·303	vvs	0·303	vvs	0·304	vvs	0·303	vvs	0·303	s	0·303	ms	0·303	ms	0·303	ms
0·249	m			0·249	w	—	—	0·250	w	—	—	—	—	—	—	—	—	—	—
0·228	s			0·228	w	—	—	—	—	—	—	0·228	w	—	—	0·228	vw	—	—
0·209	s			0·209	w	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·191	ms			0·191	w	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·187	ms			0·187	w	—	—	—	—	—	—	—	—	—	—	—	—	—	—
C-S-H																			
0·307	vs																		
0·280	s																		
0·183	s																		

pfa + 20% Lime Cured at 95°C

Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
Mullite (M)																			
0·539	vs			0·541	vw	0·539	vw	0·542	w	0·539	w	0·537	w	0·536	w	0·539	w	0·538	w
0·342	vs			0·343	w	0·342	w	0·342	w	0·342	w	0·342	w	0·342	w	0·349	w	0·343	w
0·339	vvs			0·340	w	0·339	w	0·340	w	0·340	w	0·338	w	0·339	w	0·339	ms	0·339	w
0·269	vs			0·269	w	0·269	w	0·270	w	0·271	w	0·269	w	0·269	w	0·269	w	0·269	w
0·254	vs			0·255	w	—	—	0·255	w	—	—	0·254	w	0·254	w	0·254	w	0·254	w
0·220	vs			0·221	w	—	—	—	—	—	—	0·2209	w	—	—	0·220	w	0·220	w
				—	—	—	—	—	—	—	—	0·208	vw	—	—	—	—	—	—
Quartz (Q)																			
0·426	s			—	—	0·247	w	—	—	—	—	—	—	—	—	0·426	w	0·426	w
0·334	vvs			0·335	ms	0·335	ms	0·335	ms	0·335	ms	0·335	ms	0·334	ms	0·334	ms	0·334	ms
Haemetite (Ha)																			
0·367	s			—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·269	vvs			0·269	w	0·269	w	0·270	w	0·271	w	0·269	w	0·269	w	0·269	w	0·269	w
0·251	vs			0·251	w	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·220	s			0·221	w	—	—	—	—	—	—	0·221	w	—	—	0·221	w	0·220	vw
Magnetite (Mg)																			
0·297	vs			—	—	0·289	w	—	—	0·296	w	—	—	0·295	w	—	—	—	—
0·253	vvs			—	—	—	—	0·255	w	—	—	0·254	w	—	—	—	—	—	—

Appendix 6 continued...

Appendix 7

Comparison of calculated and observed d spacings for hydrogarnet

For cubic unit cell, calculated d spacings (dc) :

$$dc = a/\sqrt{h^2+k^2+l^2}$$

therefore a = 1.2337 nm,

hkl	dc	do*
211	0.503	0.504
220	0.436	0.436
321	0.329	0.329
400	0.308	0.308
420	0.275	0.276
422	0.251	0.251
510, 431	0.241	0.242
521	0.225	0.225
611	0.200	0.200

* typical example of d spacings observed for hydrogarnet in the current work, specimen in this case is pfa + 20wt.% lime cured at 95°C and 100% r.h. for 14 days (see Appendix 6).

'Desulphurised' pfa + 20 % added lime cured at 95°c																			
Hydrogarnet (H)																			
Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
0·503	S	—	—	—	—	0·504	W	0·504	W	0·504	M	0·504	W	0·501	W	0·502	W	0·500	W
0·436	S	—	—	—	—	0·434	W	0·434	W	0·434	W	0·434	W	0·434	W	—	—	—	—
0·329	M	—	—	—	—	—	—	0·330	W	—	—	—	—	—	—	0·320	W	0·317	W
0·308	M	—	—	—	—	0·308	M	0·309	M	0·309	W	0·309	W	0·309	W	0·305	W	0·304	W
0·276	S	—	—	—	—	0·276	W	0·276	M	0·276	M	0·276	W	0·275	W	0·274	W	0·273	W
0·251	M	—	—	—	—	0·251	W	0·251	W	0·251	M	0·251	W	0·251	W	0·250	W	0·250	W
0·242	M	—	—	—	—	—	—	0·242	W	0·242	W	—	—	—	—	0·241	W	—	—
0·225	M	—	—	—	—	0·225	W	0·225	W	0·225	W	0·225	W	—	—	0·223	W	0·224	W
0·200	MS	—	—	—	—	0·200	W	0·200	VW	0·200	VW	0·199	VW	0·200	VW	0·208	VW	0·200	VW
Lime (L)																			
0,490	VS	0·488	VS	0·488	VS	0·488	M	0·488	W	0·488	W	—	—	—	—	—	—	—	—
0·311	M	0·309	W	0·309	W	0·308	W	0·309	W	0·309	W	0·309	W	—	—	—	—	—	—
0·262	VVS	0·261	VVS	0·261	VVS	0·262	S	—	—	0·262	W	0·262	W	—	—	—	—	—	—
0·192	MS	0·192	VW	0·192	VW	0·192	VW	0·192	VW	—	—	0·197	VW	—	—	—	—	—	—
0·179	MS	0·179	VW	0·179	VW	0·179	VW	—	—	0·179	VW	—	—	—	—	—	—	—	—
0·168	M	0·167	VW	0·168	VW	0·169	VW	0·169	VW	0·169	VW	0·168	VW	—	—	—	—	—	—

Appendix 8 XRD data for system D pfa cured up to 28 days.

d = d specings (nm), I = peak intensity, w = weak, s = strong, m = mild, v = very.

Desulphurised pfa+20 % Lime Cured at 95°c

Effringite (E)

Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
0·973	vvs	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·561	vs	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·498	m	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·464	ms	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·388	s	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·348	m	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·277	ms	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·256	s	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·220	s	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Mullite (M)																			
0·539	vs	0·537	w	0·538	w	0·538	w	0·538	w	0·538	w	0·537	w	0·537	w	0·538	w	0·538	w
0·342	vs	0·342	ms	0·341	ms	0·341	ms	0·341	m	0·341	m	0·342	m	0·341	ms	0·340	ms	0·340	ms
0·339	vvs	0·338	ms	0·338	ms	0·338	ms	0·338	m	0·338	ms	0·338	ms	0·339	ms	0·338	ms	0·335	ms
0·269	vs	0·271	w	0·271	w	0·269	w	0·269	vw	0·270	w	0·270	w	0·269	w	0·268	w	0·269	w
0·254	vs	0·258	w	0·254	w	0·255	w	0·254	vw	0·255	w	0·255	w	0·255	w	0·259	w	0·257	w
0·220	vs	0·220	w	0·220	w	0·220	w	0·220	vw	0·221	w	0·220	w	0·220	w	0·220	w	0·220	w
Calcite (C)																			
0·303	VVS	0·302	W	0·302	W	0·302	W	0·303	W	0·303	W	0·303	W	0·303	W	0·303	W	0·303	W
0·228	S	0·229	VW	0·229	VW	0·229	VW	0·229	VW	0·228	VW	0·229	VW	0·229	VW	0·223	VW	0·224	VW

Appendix 8 continued...

"Desulphurised" pfa + Lime Cured at 95°c

Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
Quartz (Q)																			
0·426	s	0·423	w	0·426	w	0·426	w	0·426	w	0·427	w	0·427	w	0·425	w	0·423	w	0·423	w
0·334	vvs	0·334	ws	0·334	ws	0·334	ws	0·334	ws	0·335	ws	0·334	ws	0·333	ws	0·334	ws	0·331	ws
Haemetite (Ha)																			
0·367	s	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·269	vvs	0·271	w	0·271	w	0·269	w	0·271	vw	0·270	w	0·270	w	0·269	w	0·268	w	0·269	w
0·251	vs	0·251	w	0·251	w	0·251	m	0·251	w	0·251	w	0·251	w	0·251	w	0·250	w	0·250	w
0·220	s	0·220	w	0·220	w	0·220	w	0·220	vw	0·220	w	0·220	w	0·220	w	0·220	w	0·220	w
Magnetite (Mg)																			
0·297	vs	0·292	vw	0·292	vw	0·292	vw	0·292	vw	0·292	vw	0·292	vw	0·292	vw	0·295	vw	0·297	vw
0·253	vvs	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Tobermorite (T)																			
1·100	s	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·560	ms	—	—	—	—	—	—	—	—	0·568	vw	—	—	—	—	—	—	—	—
0·307	vvs	0·302	w	0·302	w	0·302	w	0·302	w	0·303	w	0·303	w	0·303	w	0·303	w	0·303	w
0·297	s	0·292	vw	0·292	vw	0·292	vw	0·292	vw	0·292	vw	0·292	vw	0·292	vw	0·295	vw	0·297	vw
0·280	s	0·288	w	0·288	w	0·288	w	0·288	w	0·288	w	0·288	w	0·288	w	0·287	w	0·287	w
0·228	ms	0·229	vw	0·229	vw	0·229	vw	0·229	vw	0·229	vw	0·229	vw	0·229	vw	0·223	vw	0·224	vw
0·183	vs	0·183	w	0·183	w	—	—	0·183	w	0·183	w	0·183	w	0·183	w	0·181	w	—	—

Appendix 8 continued...

pfa +20% Lime + 4% Gypsum Cured at 95°c

Hydrogarnet (H)																			
Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
0·503	s	—	—	—	—	—	—	0·504	w	0·506	w	0·505	w	0·506	w	0·502	w	0·502	w
0·436	s	—	—	—	—	—	—	—	—	0·437	w	—	—	0·437	w	0·435	w	0·435	w
0·329	m	—	—	—	—	—	—	—	—	—	—	0·331	w	0·331	w	0·323	w		w
0·308	m	—	—	—	—	—	—	—	—	0·309	w	0·309	w	0·309	w	0·308	w	0·308	w
0·276	s	—	—	—	—	—	—	0·277	w	0·277	w	0·276	w	0·277	w	0·276	w	0·276	w
0·251	m	—	—	—	—	—	—	—	—	0·250	w	—	—	—	—	—	—	—	—
0·242	m	—	—	—	—	—	—	—	—	0·243	w	0·243	w	—	—	0·242	w	—	—
0·225	m	—	—	—	—	—	—	0·226	w	0·226	w	0·226	w	0·226	w	0·225	w	0·225	w
0·200	ms	—	—	—	—	—	—	0·200	vw	0·201	vw	0·201	vw	0·201	vw	0·200	vw	0·200	vw
Lime (L)																			
0·490	vs	0·492	vs	0·489	vs	0·488	vs	0·489	m	—	—	—	—	—	—	—	—	—	—
0·311	m	0·310	m	0·310	m	0·310	m	—	—	—	—	—	—	—	—	—	—	—	—
0·262	ws	0·262	vs	0·262	vs	0·261	vs	0·263	m	—	—	—	—	—	—	—	—	—	—
0·192	ms	0·192	m	0·192	w	0·192	w	0·192	vw	—	—	—	—	—	—	—	—	—	—
0·179	ms	0·179	m	0·179	w	0·179	w	0·180	vw	—	—	—	—	—	—	—	—	—	—
0·168	m	—	—	0·168	vw	0·168	w	0·168	vw	—	—	—	—	—	—	—	—	—	—

Appendix 9 XRD data for system 2 cured up to 28 days.

d = d specings (nm), I = peak intensity, w = weak, s = strong, m = mild, v = very.

pfa + 20% Lime + 4% Gypsum Cured at 95 °c

Ettringite (E)

Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
0·973	ws	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·561	vs	0·577	m	0·557	m	0·557	s	0·560	ms	0·562	w	0·563	w	0·563	w	0·558	w	—	—
0·498	m	—	—	—	—	—	—	0·496	w	—	—	—	—	—	—	—	—	—	—
0·464	ms	—	—	0·466	w	0·466	w	0·469	w	0·471	w	0·470	w	—	—	—	—	—	—
0·388	s	0·386	m	0·386	m	0·386	ms	0·387	m	0·388	m	0·389	w	0·389	w	0·387	w	—	—
0·348	m	—	—	0·346	w	0·346	w	0·347	w	—	—	—	—	—	—	—	—	—	—
0·321	m	0·321	w	—	—	0·322	w	—	—	—	—	—	—	—	—	—	—	—	—
0·277	ms	—	—	0·276	w	0·276	w	0·277	w	0·277	w	0·276	w	0·277	w	0·276	w	0·276	w
0·256	s	0·255	w	0·255	w	0·255	w	0·256	w	0·256	w	0·257	w	0·255	w	0·254	w	0·254	w
0·220	s	0·223	w	0·224	w	0·220	w	0·225	w	0·226	w	0·226	w	0·226	w	0·225	w	0·225	w
Gypsum (G)																			
0·756	vs	—	—	—	—	—	—	—	—	—	—	—	—	0·764	m	—	—	0·757	w
0·427	s	—	—	—	—	—	—	—	—	—	—	—	—	0·429	m	0·426	m	0·426	m
0·379	m	—	—	—	—	—	—	—	—	—	—	—	—	0·381	w	—	—	—	—
0·305	s	—	—	—	—	—	—	—	—	—	—	—	—	0·306	m	0·308	m	0·308	m
0·286	ms	—	—	—	—	—	—	—	—	—	—	—	—	0·290	w	—	—	0·289	w
0·267	ms	—	—	—	—	—	—	—	—	—	—	—	—	0·270	w	0·269	w	0·269	w

pfa+20% Lime+4% Gypsum Cured at 95°c

Standered		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
Bassanite (B)																			
0·598	vs	—	—	—	—	—	—	—	—	—	—	—	—	—	—	0·575	w	—	—
0·345	s	—	—	—	—	—	—	—	—	—	—	—	—	—	—	0·349	m	0·349	m
0·298	vvs	—	—	—	—	—	—	—	—	—	—	—	—	—	—	0·300	w	—	—
0·278	vvs	—	—	—	—	—	—	—	—	—	—	—	—	—	—	0·284	w	—	—
0·212	s	—	—	—	—	—	—	—	—	—	—	—	—	—	—	0·209	w	—	—
Mullite (M)																			
0·539	vs	0·538	w	0·537	w	0·536	w	0·538	w	0·541	w	0·541	w	0·541	w	0·538	w	0·537	w
0·342	vs	0·342	ms	0·342	ms	0·346	ms	0·342	ms	0·343	ms	—	—	0·344	ms	0·342	ms	0·342	ms
0·339	vvs	0·339	ms	0·338	ms	0·338	ms	0·339	ms	0·340	ms	0·340	ms	0·340	ms	0·339	ms	0·339	ms
0·269	vs	0·269	w	0·269	w	0·269	w	0·270	w	0·270	w	0·270	w	0·270	w	0·269	w	0·269	w
0·254	vs	0·255	w	0·255	w	0·255	w	0·256	w	0·256	w	0·255	w	0·255	w	0·254	w	0·254	w
0·220	vs	0·220	w	0·220	w	0·220	w	0·220	w	0·221	w	0·221	w	0·221	w	0·220	w	0·221	w
Quartz (Q)																			
0·426	s	0·427	w	0·426	w	0·426	w	0·426	w	0·427	w	0·427	w	0·429	w	0·426	w	0·426	w
0·334	vvs	0·334	vs	0·334	vs	0·334	vs	0·335	vs	0·336	vs	0·336	vs	0·336	vs	0·334	vs	0·334	vs
Haemetite (Ha)																			
0·367	s	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·269	vvs	0·269	w	0·269	w	0·269	w	0·270	w	0·270	w	0·270	w	0·270	w	0·269	w	0·269	w
0·251	vs	—	—	0·251	w	0·251	w	0·252	w	0·252	w	0·252	w	0·252	w	0·252	w	0·252	w
0·220	s	0·220	w	0·220	w	0·220	w	0·220	w	0·221	w	0·221	w	0·221	w	0·220	w	0·221	w

Appendix 9 continued...

pfa + 20 % Lime + 6 % Gypsum Cured at 95°c

Hydrogarnet (H)

Standered		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
0·503	s			—	—	—	—	—	—	0·505	m	0·506	m	0·506	m	0·505	w	0·500	w
0·436	s			—	—	—	—	—	—	0·437	m	0·437	w	0·438	m	—	—	0·436	w
0·329	m			—	—	—	—	—	—	0·325	m	—	—	0·323	m	0·323	w	—	—
0·300	s			—	—	—	—	—	—	0·305	s	0·304	s	0·306	ms	0·306	m	0·307	m
0·276	m			—	—	—	—	—	—	0·276	m	0·276	m	0·276	ms	0·277	m	0·275	m
0·251	s			—	-	—	—	—	—	0·252	m	0·252	w	0·252	m	0·252	w	0·252	w
0·242	m			—	—	—	—	—	—	0·242	vw	0·241	vw	0·242	vw	0·242	vw	0·242	vw
0·225	m			—	—	—	—	—	—	0·325	m	0·225	vw	0·225	vw	0·226	vw	0·225	vw
0·200	ms			—	—	—	—	—	—	0·200	vw	0·199	vw	0·171	vw	0·200	vw		
Lime (L)																			
0·490	vs			0·492	s	0·492	s	0·492	s	0·497	m	—	—	—	—	—	—	—	—
0·311	m			0·300	m	0·311	m	0·311	m	0·309	m	—	—	—	—	—	—	—	—
0·262	v vs			0·262	s	0·262	vs	0·263	vs	0·262	w	—	—	—	—	—	—	—	—
0·192	ms			0·192	w	0·193	w	0·192	w	0·194	vw	—	—	—	—	—	—	—	—
0·179	ms			0·179	w	0·179	vw	0·179	w	0·180	vw	—	—	—	—	—	—	—	—
0·168	m			0·168	vw	0·168	vw	0·168	vw	0·167	vw	—	—	—	—	—	—	—	—

Appendix 10 XRD data for system 3 cured up to 28 days.

d = d specings (nm), I = peak intensity, w = weak, s = strong, m = mild, v = very.

pfa+20% Lime+6% Gypsum Cured at 95°c

Ettringite (E)																			
Standered		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
0·973	vvs			—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·561	vs			0·561	m	0·560	m	0·562	m	0·564	m	0·560	m	—	—	—	—	—	—
0·498	m			0·492	s	0·498	s	0·492	s	0·497	m	—	—	—	—	—	—	—	—
0·388	s			0·387	m	0·389	ms	0·387	ms	0·388	ms	0·388	ms	0·388	m	0·389	m	—	—
0·348	m			—	—	0·347	m	—	—	0·347	m	0·346	m	—	—	—	—	—	—
0·321	m			—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
0·227	ms			0·276	w	0·277	w	0·278	w	0·276	m	0·276	m	0·276	ms	0·277	m	0·275	m
0·256	s			0·252	w	0·252	w	0·255	w	0·255	w	0·254	w	0·255	w	—	—	—	—
0·220	s			0·221	w	0·221	vw	0·221	w	0·221	w	0·220	w	0·221	vw	0·221	vw	—	—
Gypsum (G)																			
0·756	vs			0·761	ws	0·761	ws	0·762	ws	0·762	ms	0·762	ms	—	—	—	—	—	—
0·427	s			0·429	s	0·428	s	0·429	vs	0·427	m	0·426	m	—	—	—	—	—	—
0·379	m			0·381	s	0·380	ms	0·380	ms	0·380	m	0·376	m	—	—	—	—	—	—
0·305	s			0·307	s	0·306	ws	0·307	s	0·305	s	0·304	s	—	—	—	—	—	—
0·286	ms			0·288	w	0·287	w	0·289	w	0·288	w	0·288	w	0·286	w	0·286	w	—	—
0·267	ms			0·270	m	0·270	w	0·270	w	0·269	w	0·270	w	—	—	—	—	—	—

Appendix 10 continued...

pfa +20 % Lime + 6 % Gypsum Cured at 95 °c

Calcite (Ca)

Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
0·303	vvs			0·303	s	—	—	—	—	0·305	s	0·304	s	0·306	ms	0·306	ms	0·304	m
0·228	s			0·229	vw	0·229	vw	0·228	vw	0·229	vw	0·228	vw	0·229	vw	0·228	vw	—	—

Unidentified Peaks:

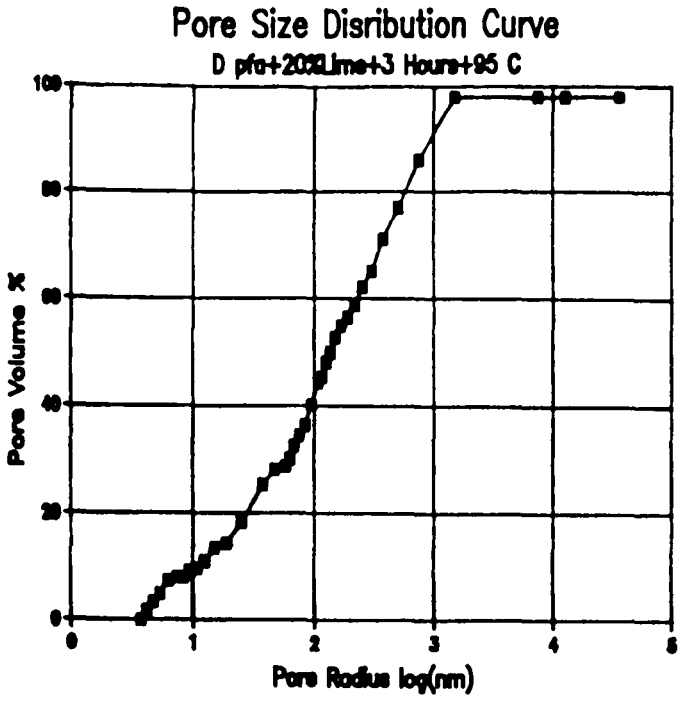
				—	—	—	—	—	—	0·713	m	0·702	m	—	—	—	—	—	—
				—	—	—	-	—	—	0·685	m	—	—	0·643	m	0·670	m	—	—
				—	—	0·651	w	—	—	—	—	—	—	—	—	—	—	—	—
				—	—	0·609	w	—	—	0·604	w	0·590	w	0·610	w	0·619	w	—	—
				—	—	0·460	w	-	—	—	—	—	—	—	—	—	—	—	—
				—	—	0·449	w	—	—	—	—	—	—	—	—	—	—	—	—
				0·399	m	—	—	0·408	m	0·408	w	—	—	—	—	—	—	—	—
				—	—	-	—	—	—	—	-	0·358	m	0·351	ms	0·351	ms	0·351	m
				—	—	—	—	0·325	m	0·325	m	—	—	—	—	—	—	-	—

Appendix 10 continued...

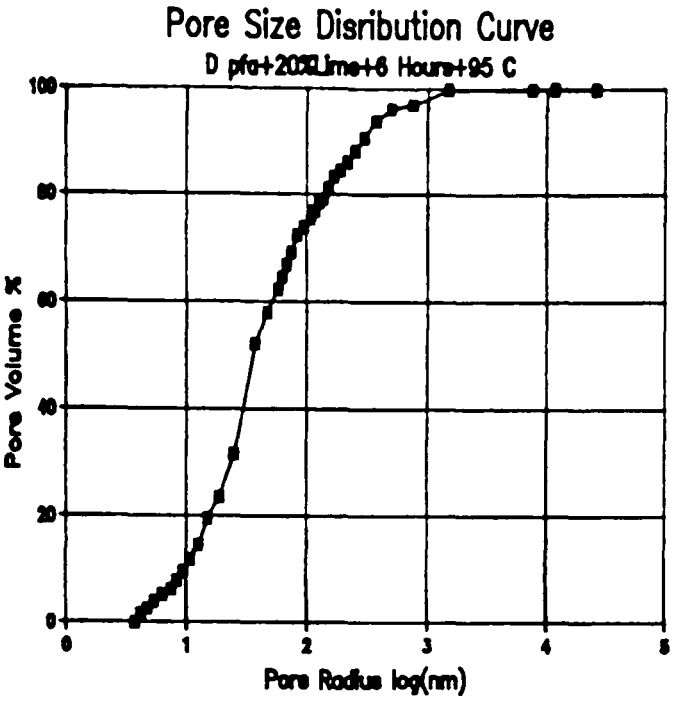
pfa+20% Lime+6% Gypsum Cured at 95°c

Mullite (M)																			
Standard		3 Hours		6 Hours		12 Hours		18 Hours		24 Hours		3 Days		7 Days		14 Days		28 Days	
d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I	d	I
0·539	vs			0·539	m	0·540	m	0·543	m	0·541	m	0·540	m	0·541	m	0·538	—	0·538	w
0·342	vs			0·343	ms	0·343	ms	0·343	ms	0·343	ms	—	—	—	—	0·340	ms	0·342	ms
0·339	vvs			0·339	s	0·339	ms	0·341	s	0·340	ms	0·340	ms	0·340	ms	—	—	—	—
0·269	vs			0·270	m	0·270	w	0·270	w	0·269	w	0·270	w	0·270	m	0·270	m	0·269	w
0·254	vs			0·252	w	0·252	w	0·252	w	0·252	m	0·254	w	0·252	w	0·252	w	0·253	w
0·220	vs			0·221	w	0·221	vw	0·221	w	0·221	w	0·220	w	0·221	vw	0·221	vw	0·221	vw
Quartz (Q)																			
0·426	s			0·429	s	0·428	s	0·429	s	0·427	m	0·426	m	0·427	m	0·426	m	0·426	m
0·334	ws			0·336	vs	0·335	vs	0·336	s	0·336	s	0·336	s	0·336	s	0·337	s	0·336	s
Haemetite (Ha)																			
0·367	s			0·360	m	0·362	m	0·366	m	—	—	—	—	0·360	m	0·361	m	—	—
0·269	vvs			0·270	m	0·270	w	0·270	w	0·269	w	0·270	w	0·270	m	0·270	m	0·269	w
0·251	vs			0·252	w	0·252	w	0·252	w	0·258	m	0·252	w	0·252	m	0·252	w	0·252	w
0·220	s			0·221	w	0·221	vw	0·221	w	0·221	w	0·220	w	0·221	vw	0·221	vw	0·221	vw
Magnetite (Mg)																			
0·297	vs			0·295	w	0·296	w	0·297	m	0·295	m	0·296	m	—	—	—	—	—	—
0·253	vvs			0·252	w	0·252	w	0·255	w	0·252	w	0·252	w	0·252	w	0·252	w	0·252	w
0·242	ms			—	—	—	—	0·242	vw	0·241	vw	0·242	vw	0·242	vw	0·242	vw	0·242	vw

Appendix 10 continued...



a



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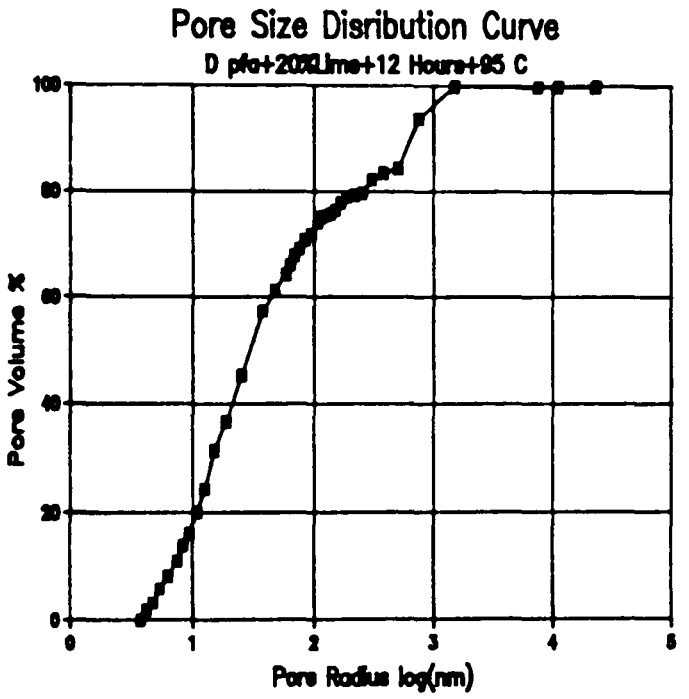

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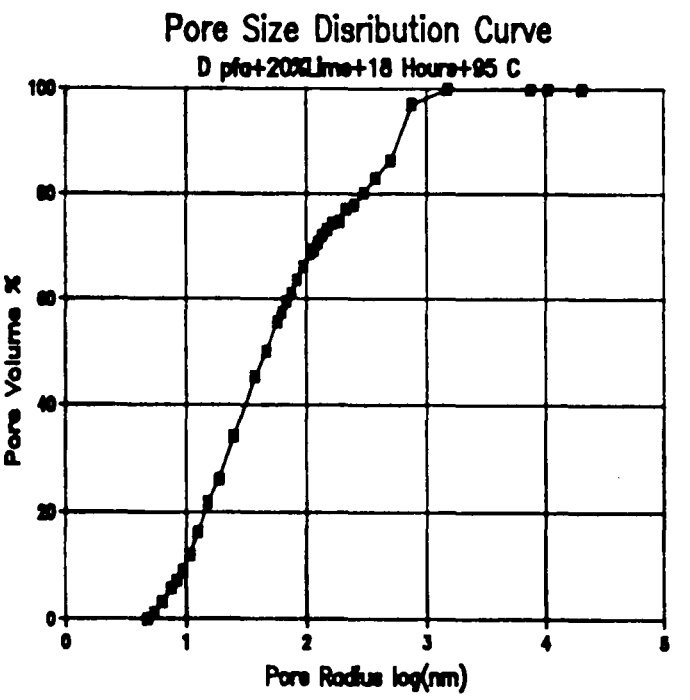

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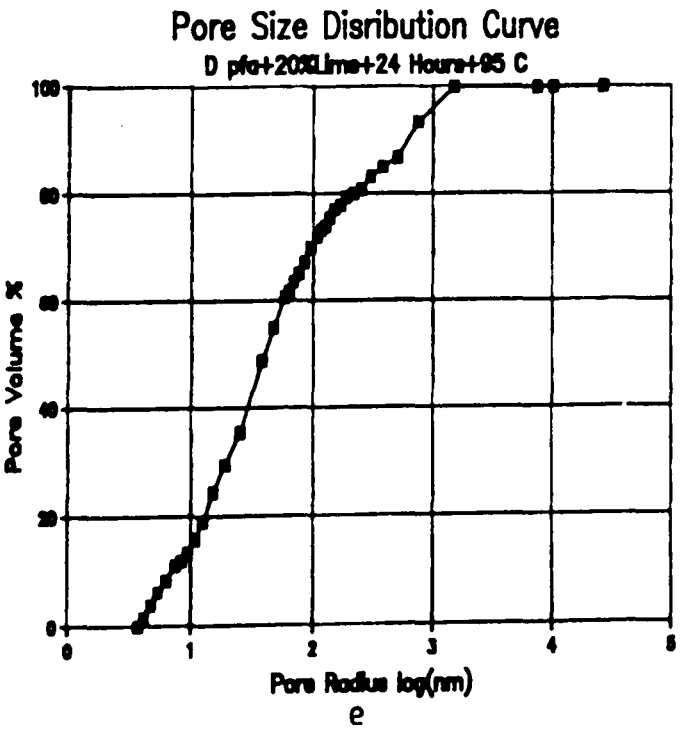

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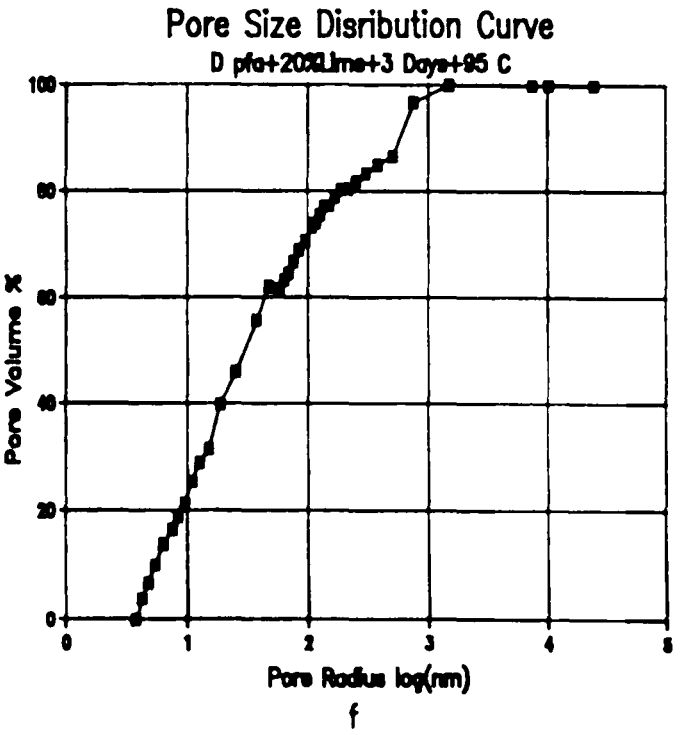
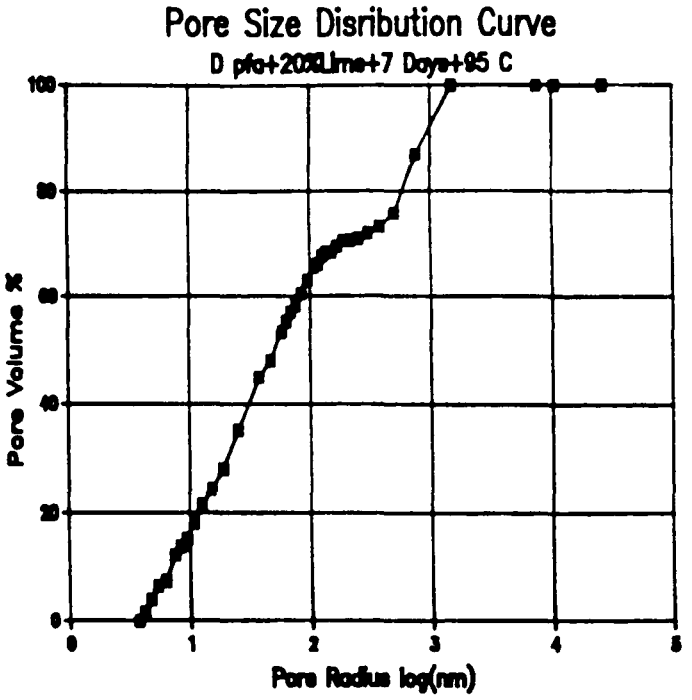
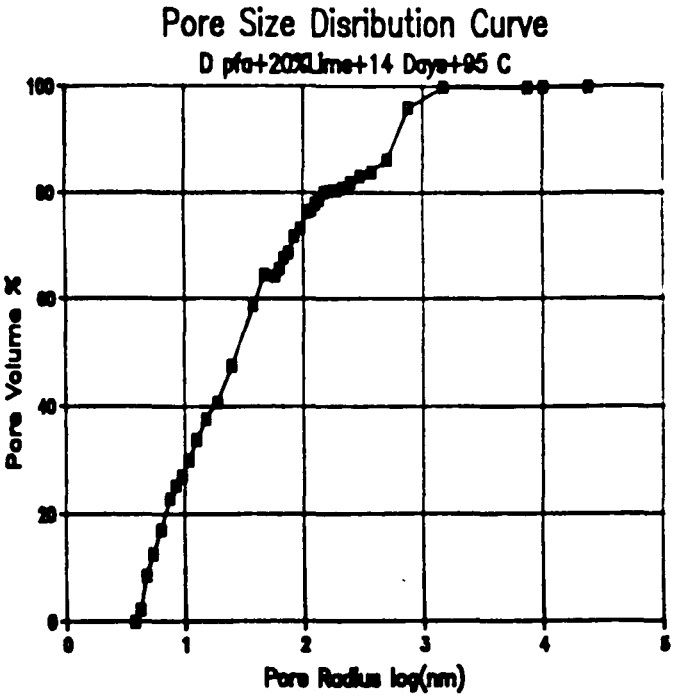


f

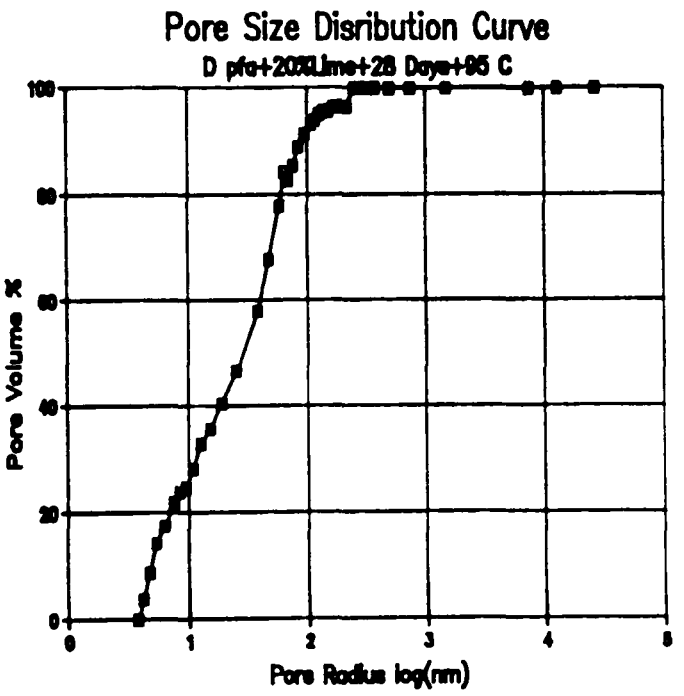
Appendix 11a-i Plots of cumulative pore volume (expressed as a percentage of total porosity) versus the logarithm of pore radius for system D pfa samples cured at 95°C for curing times of between 3 hours and 28 days.



g

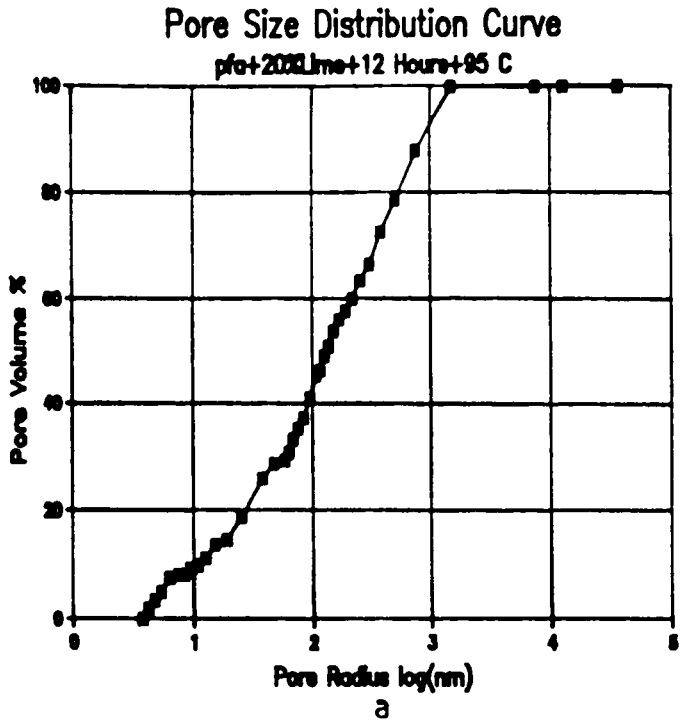


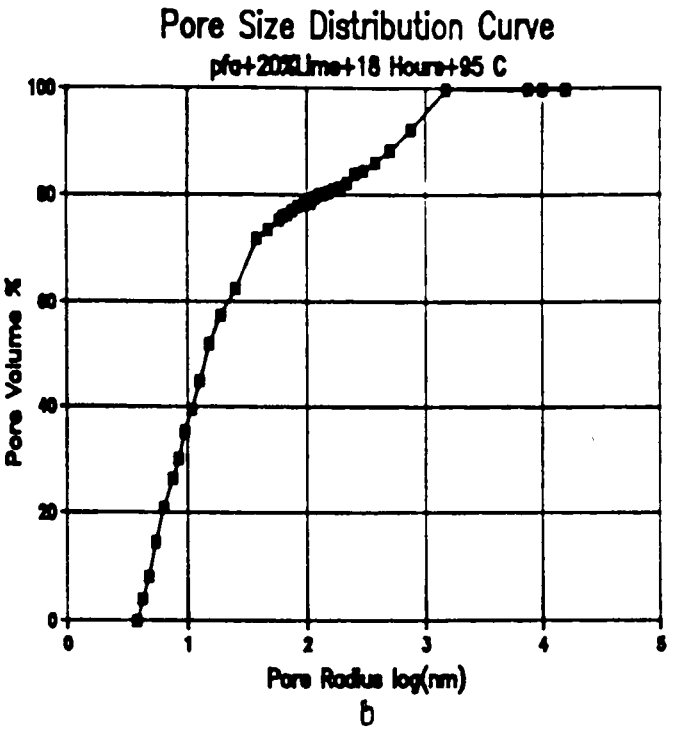
h

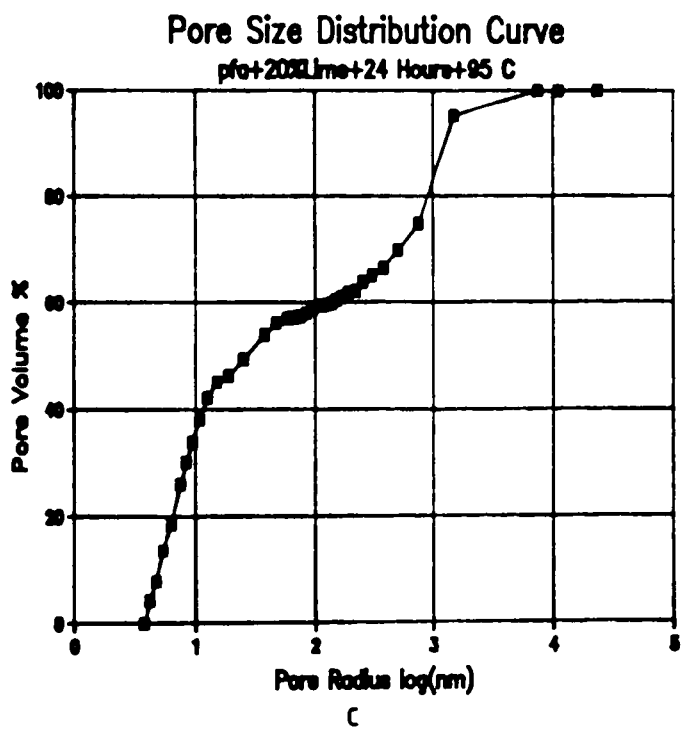


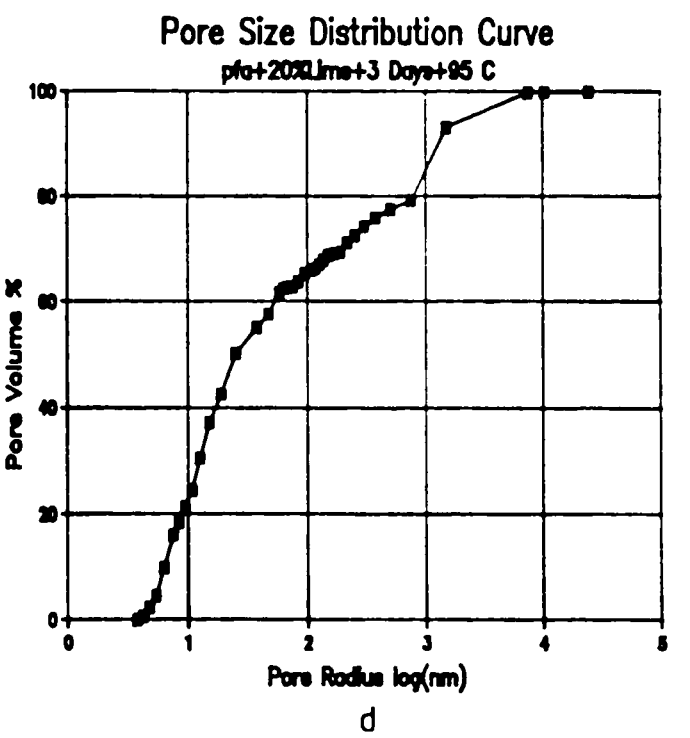
i

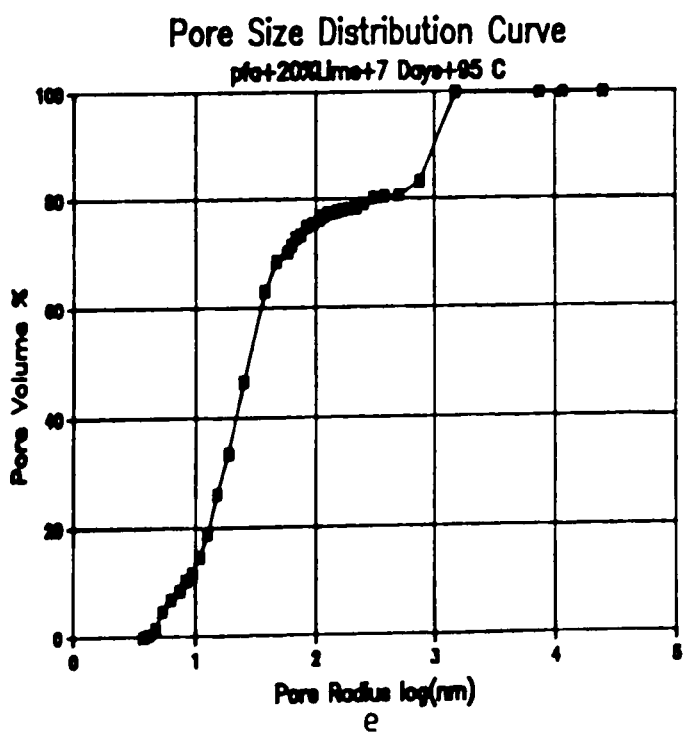
Appendix 11a-i continued...

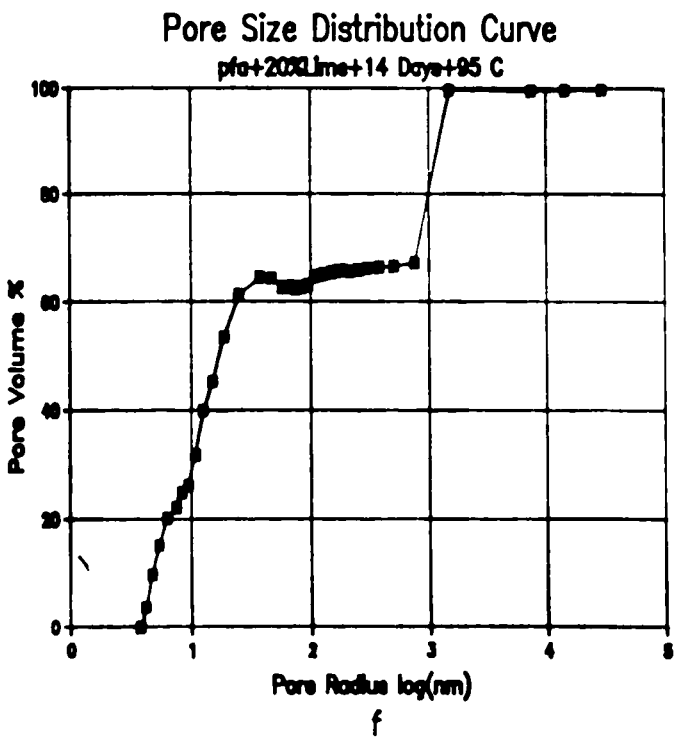




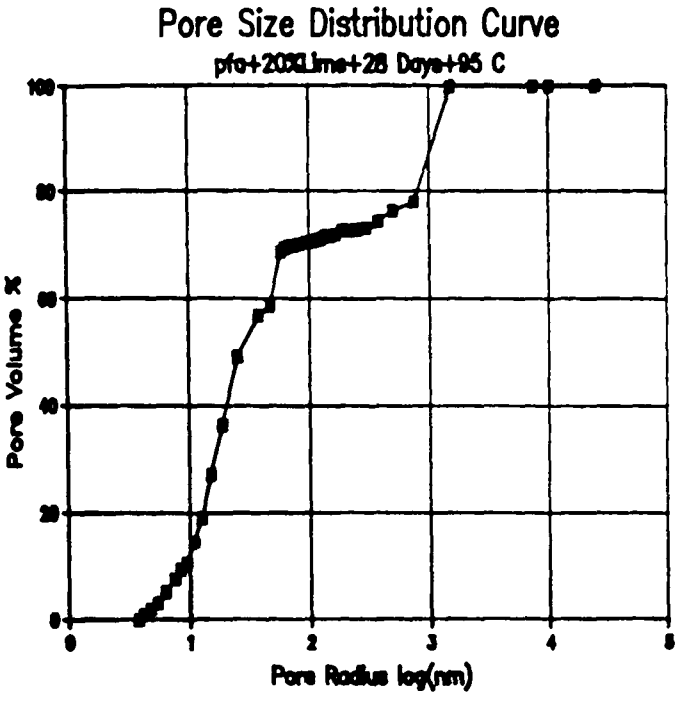






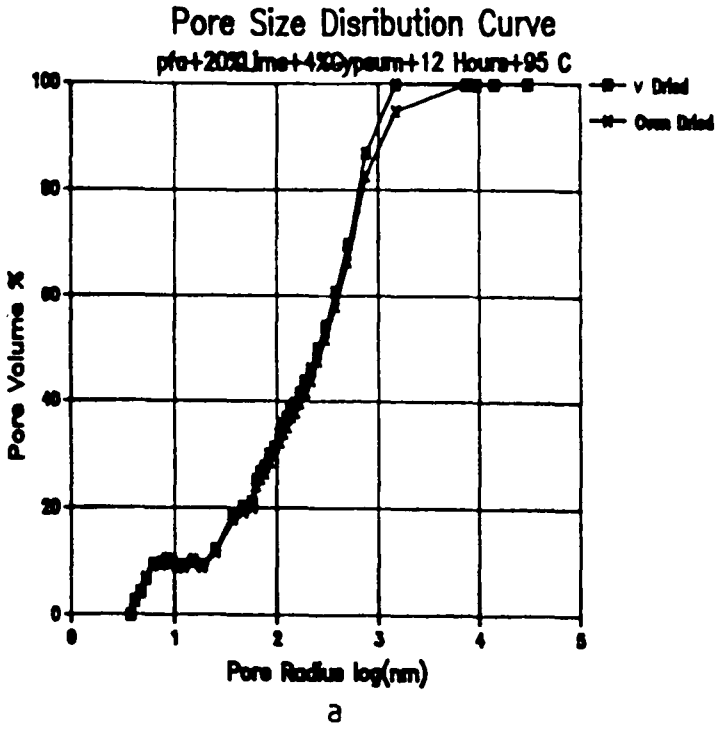


Appendix 12a-g Plots of cumulative pore volume (expressed as a percentage of total porosity) versus the logarithm of pore radius for system 1 samples cured at 95°C for curing times of between 12 hours and 28 days.

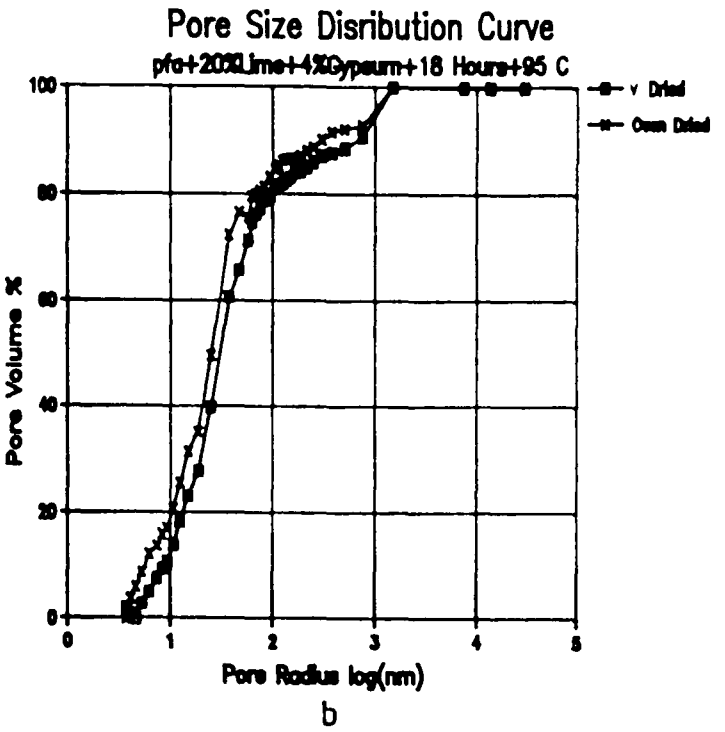


g

Appendix 12a-g continued...



a



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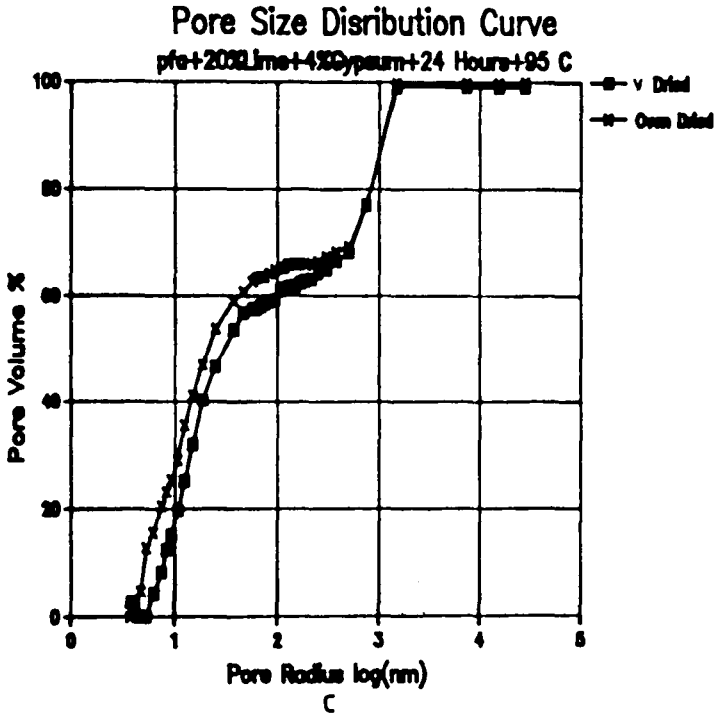

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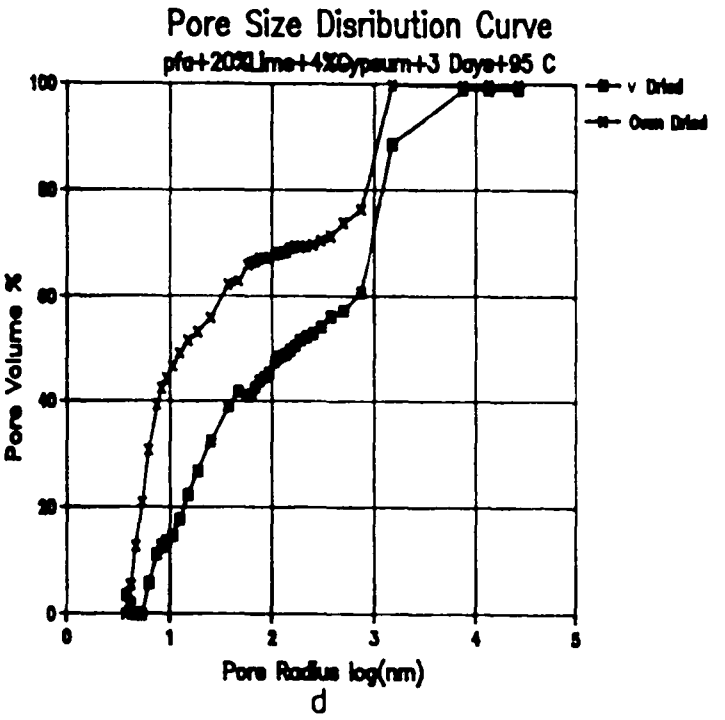

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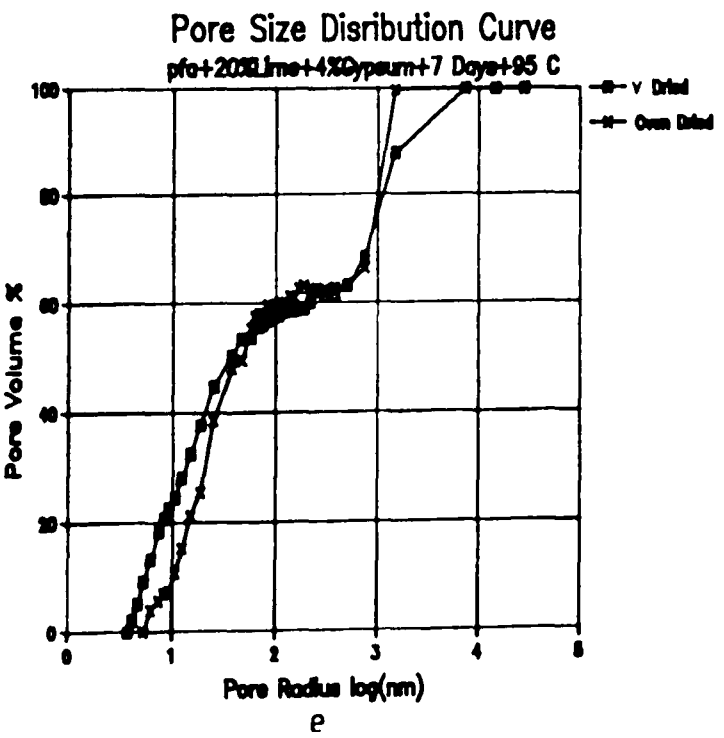

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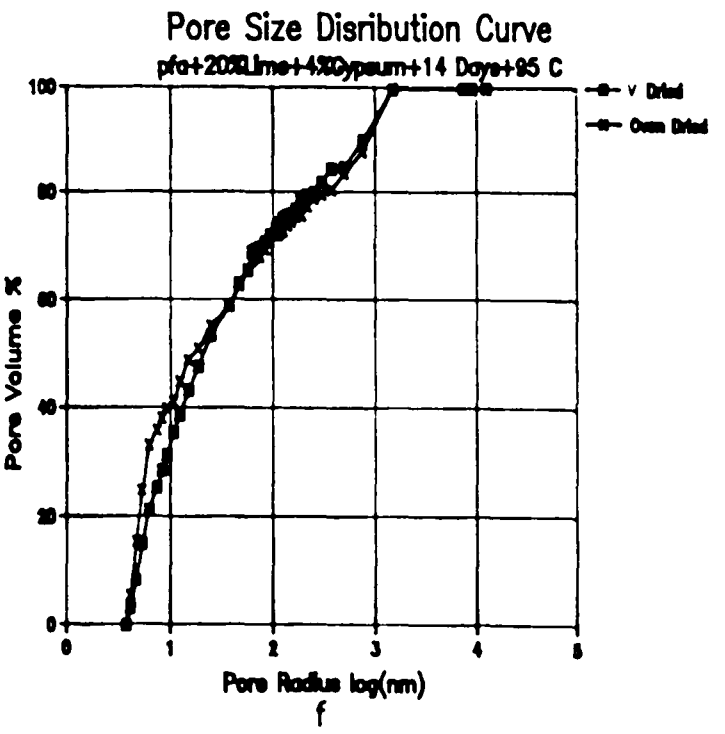
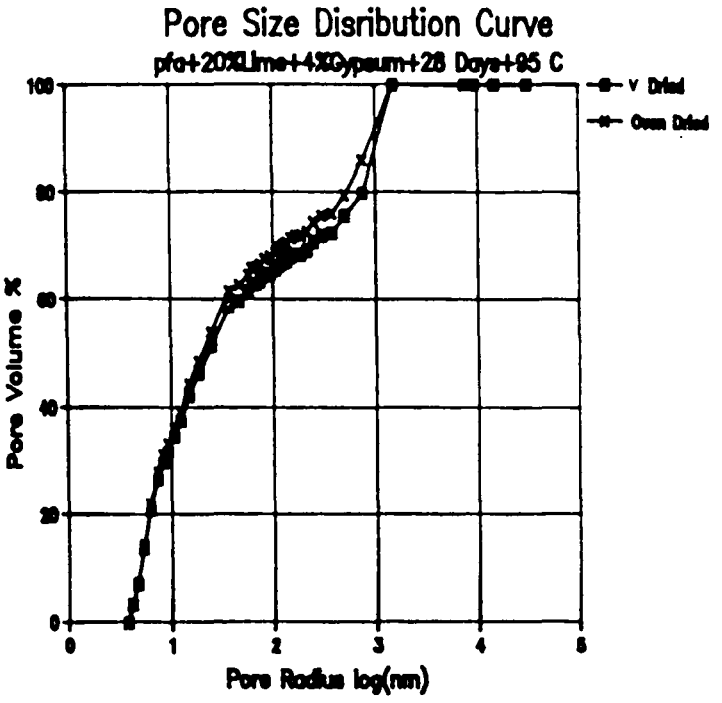


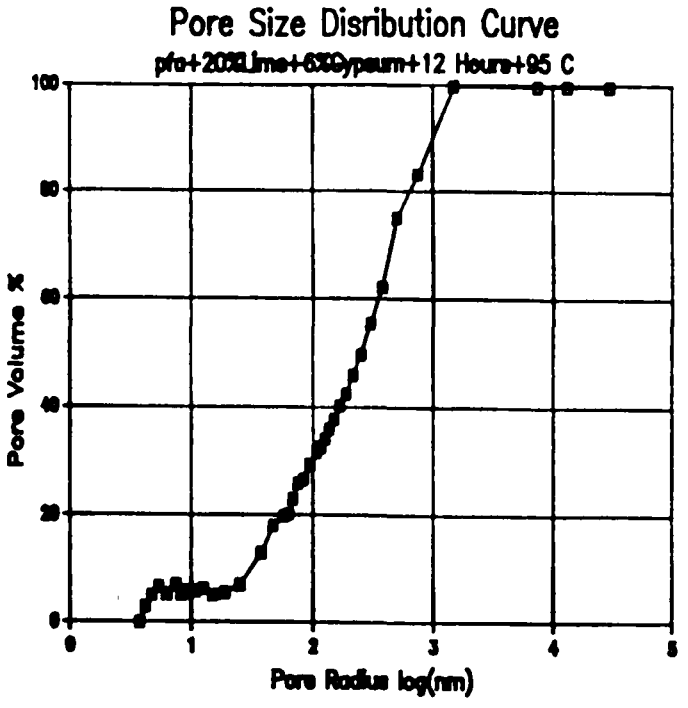
f

Appendix 13a-g Plots of cumulative pore volume (expressed as a percentage of total porosity) versus the logarithm of pore radius for system 2 samples cured at 95°C for curing times of between 12 hours and 28 days.

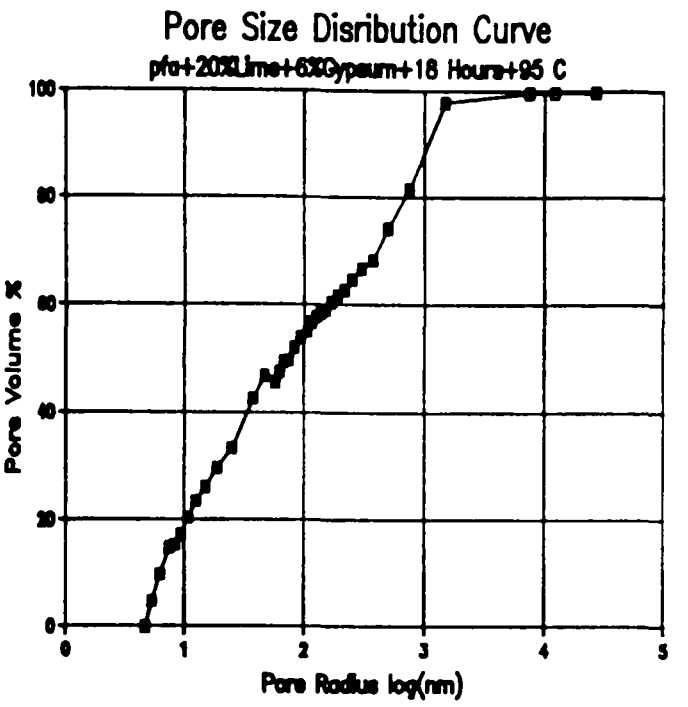


g

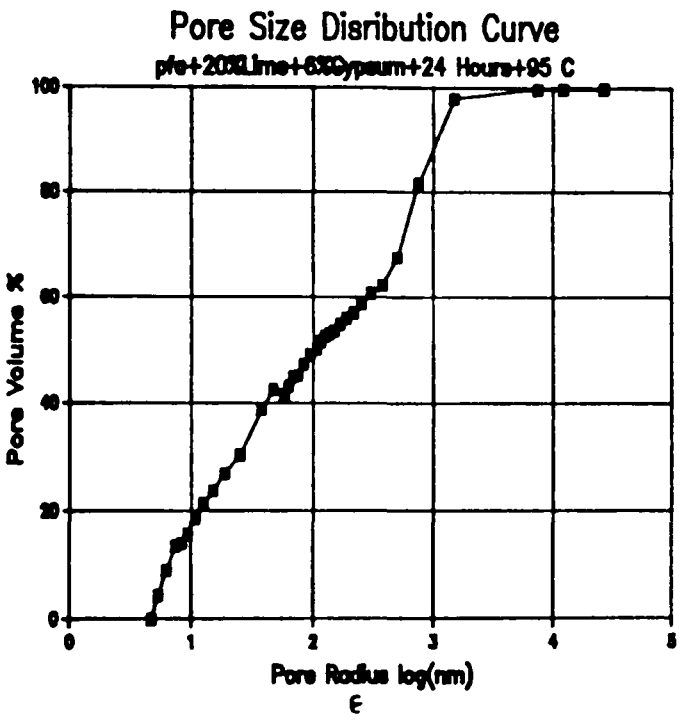
Appendix 13a-g continued...



a



b



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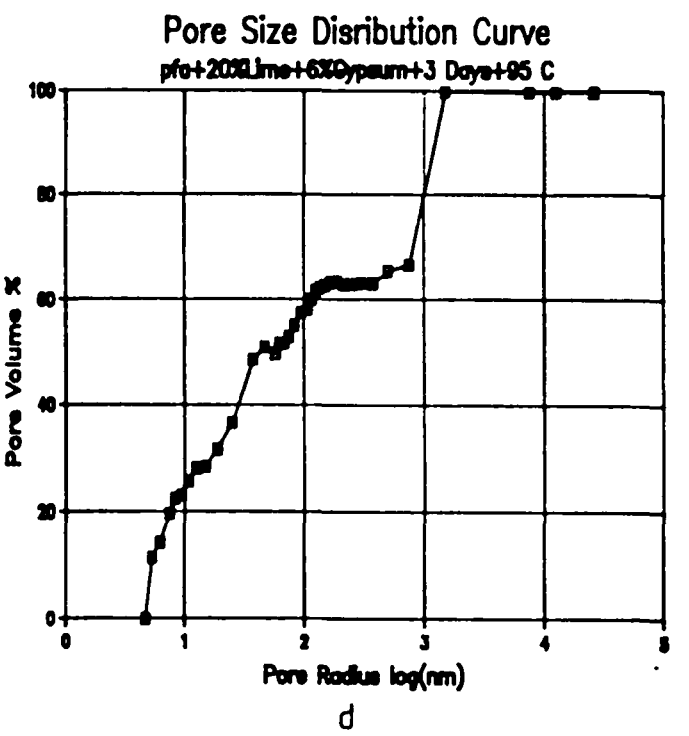

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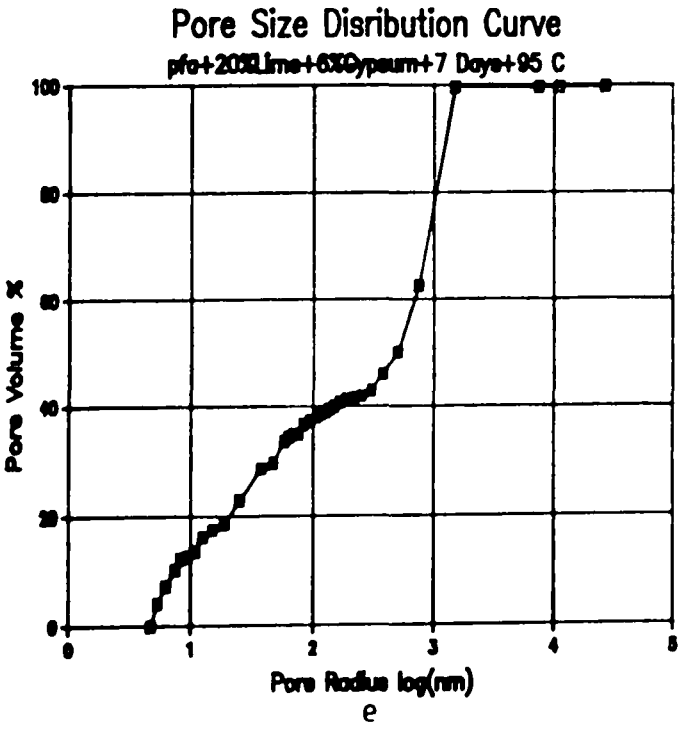

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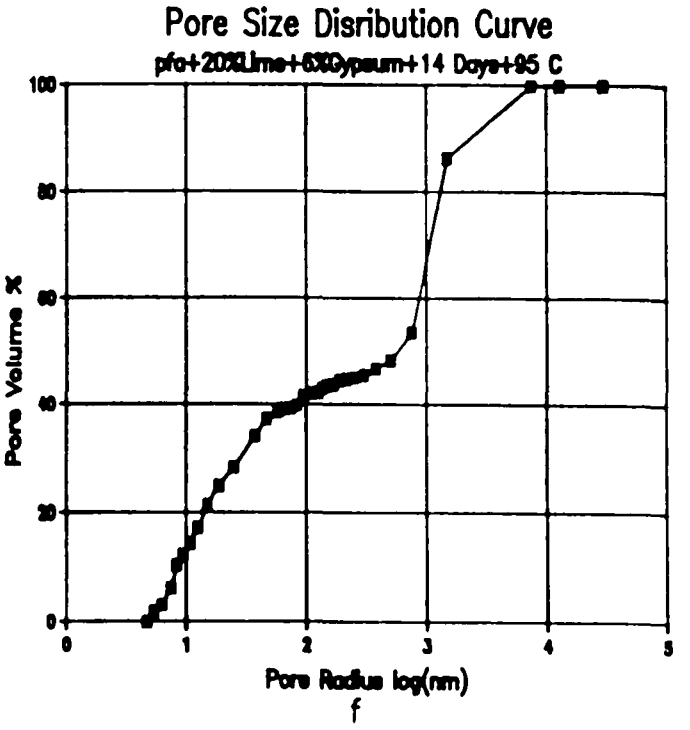
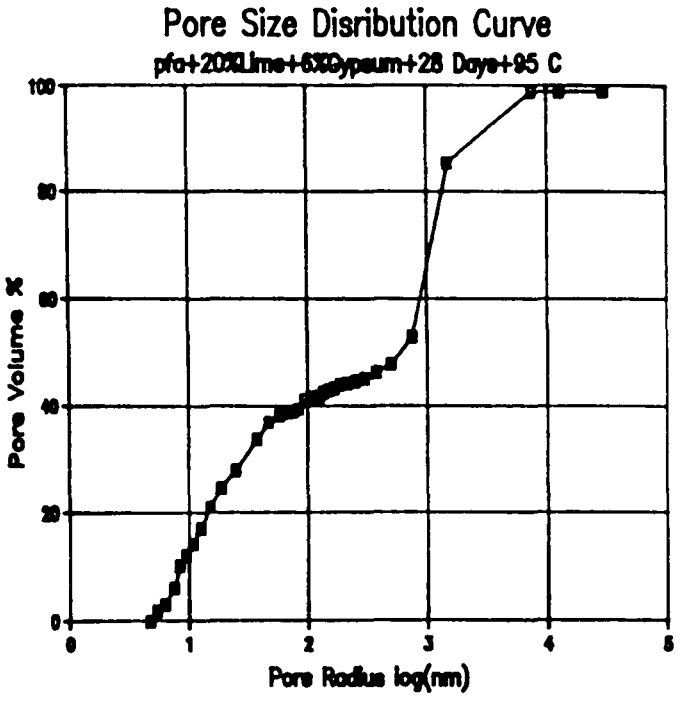


f

Appendix 14a-g Plots of cumulative pore volume (expressed as a percentage of total porosity) versus the logarithm of pore radius for system 3 samples cured at 95°C for curing times of between 12 hours and 28 days.



g

Appendix 14a-g continued...

Appendix 15

Method of calculating the pore volumes of green pfa-lime-gypsum cylinders

Prior to any reaction taking place the pore space between the solid particles consists of water filled space and entrapped air. On drying this water is lost.

Let
Wt = dry weight of the sample
Vt = bulk volume of the sample
Vs = volume of the solid materials in the sample
Vp = volume of the pores in the sample
then,
the pore volume per unit mass of solid Pv = Vp/Wt,
the bulk volume Vt = Vs + Vp,
the bulk density = Wt/Vt, and
the solid material density = Ws/Vs
Therefore

$$Vp/Wt = (Vt-Vs)/Wt = Vt/Wt - Vs/Wt = (1/Pd) - (1/Ps) \quad \text{eq. 1}$$

Hence

$$Pv = (1/Pd) - (1/Ps) \quad \text{eq. 2}$$

Hence by calculating the solid material density (from the densities of the component solids) and determining the dry density of the sample, the initial pore volume per unit mass of solid can be calculated.